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Degradation rates and ageing effects of UV on tyre and road wear particles

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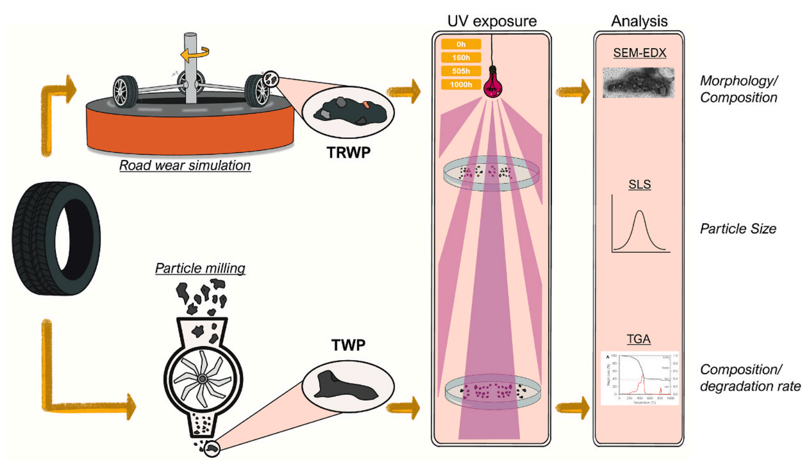
HIGHLIGHTS

- UV degradation of tyre wear is a first order process with a rate of 0.03 day^{-1}.
- Modal particle size is reduced by 0.03 μm day^{-1}.
- A fraction of smaller tyre wear particles <10 μm is formed through fragmentation.
- Environmental measurements confirm influence of degradation of accumulation.
- Smaller tyre wear particles observed in soil than atmospheric deposition samples.

GRAPHICAL ABSTRACT



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ABSTRACT

Tyre and road wear particles (TRWPs) are estimated to be the largest source of microplastics in the environment and due to the intrinsic use of tyres in our society this will continue to grow. Understanding their degradation mechanisms and subsequent accumulation over time is important to gain insights into the fate and impact of these particles in the environment. Accelerated UV-ageing was performed on cryomilled tyre tread particles and TRWPs from a road simulator to investigate the abiotic degradation of rubber. Degradation was followed with thermogravimetric analysis (TGA) that led to an average abiotic degradation rate of 0.025 day^{-1} when corrected for the acceleration factor. Static light scattering (SLS) showed that during degradation, the average particle size reduced by 0.03 μm day^{-1} and smaller particles <10 μm were formed. Further characterisation with scanning electron microscopy (SEM) and energy dispersive x-ray spectroscopy (EDX) confirmed these findings and showed that the sulphur content is reduced through UV-ageing suggesting that crosslinking breakage may be a mechanism of degradation. Analysis with gas chromatography and mass spectrometry (GC-MS) showed a substantial decrease in chemical additives by UV-induced oxidation and breakdown. Finally, with measurements in the field

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TRWP particle sizes and accumulation times were studied, confirming the experimentally determined degradation mechanisms.

Abbreviations and Units

BR	Butadiene Rubber
BSE	Back Scattered Electrons
CB	Carbon Black
DTG	Differential Thermogravimetry
EC	Elemental Carbon
EDX	Energy-dispersive X-ray spectroscopy
LN_2	Liquid Nitrogen
NR	Natural Rubber
PM	Particulate Matter
SBR	Styrene Butadiene Rubber
SE	Secondary Electrons
SEM	Scanning Electron Microscopy
SEM-EDX	Scanning Electron Microscopy with Energy-Dispersive X-ray spectroscopy
SLS	Static Light Scattering
SVOC	Semi-Volatile Organic Compounds
TGA	Thermogravimetric analysis
T(R)WP	Tyre (and Road) Wear Particles
TWP	Tyre Wear Particles

1. Introduction

Tyre and road wear particles (TRWPs) are an environmental contaminant of high concern due to their high abundance and wide environmental distribution (Rødland et al., 2022a, 2022b; Son and Choi, 2022; Baensch-Baltruschat et al., 2020a; Schwarz et al., 2023; Jan et al., 2017). They are predicted to be the largest source of microplastics with annual global emissions estimated to be between 800 and 3300 kt (Schwarz et al., 2023; Jan et al., 2017). TRWPs are a form of non-exhaust vehicle emissions (NEE) that have been recognized as a growing problem and are expected to be regulated in upcoming Euro 7 legislation. TRWPs are generally described as elongated particles with a heterogeneous composition of tyre tread with encrustations of traffic related particles such as brake and road wear (Kovochich et al., 2021; Baensch-Baltruschat et al., 2020a; Kreider et al., 2010; Knight et al., 2020; Liu et al., 2021). A typical tyre tread consists of approximately 40–60% synthetic and natural rubber polymers, 20–45% reinforcing/filler agents (i.e black carbon, silica), and 5–15% chemical additives (i.e. softeners, vulcanisation agents, antioxidants, extender oils) (Giechaskiel et al., 2024a).

Several factors affect the environmental fate of TRWPs. Transport is affected by their size: small particles will be dispersed in the air, whilst large particles will deposit on or near the road surface after which they are further transported through resuspension and run-off to road side soils, sewers and surface waters (Baensch-Baltruschat et al., 2021; Jan et al., 2017). According to Baensch-Baltruschat et al., 66–76% of the coarse 'non-airborne' fraction is transported to soils and road banks near roads and 12–20% is released to surface waters (Baensch-Baltruschat et al., 2021). This is comparable with estimates in the Netherlands showing that 6% is directly ending up in surface waters, 15% in sewers and 67% in soil (Van et al., 2014), and a Swiss study estimated that 74% is deposit in roadside soils and 22% in surface waters (Sieber et al., 2020). As the most TRWPs are estimated to end up in roadside soils, understanding environmental fate of TRWPs in this compartment is of great importance. However, limited information is available on the degradation of TRWP in soils (Baensch-Baltruschat et al., 2021; Jan et al., 2017).

Often modelling studies are used to better understand the environmental fate and transport of TRWPs (Cadle and Williams, 1980; Unice et al., 2019a). An important variable in these models is how fast TRWPs degrade in the different environmental compartments. Both photo- and biodegradation are relevant degradation paths to consider. Cadle and Williams studied the environmental degradation of T(R)WPs in soil including factors such as oxygen, heat, humidity, light and microorganisms in real-time, using an open set-up exposed to the elements (Cadle and Williams, 1980). They found that degradation of tyre wear in soil had a rate of 0.15% per day and noted that the presence of microbes and type of tyre material (tread vs wear) play a big role in degradation. For unvulcanised rubber they conclude that oxidation processes are more significant than microbial degradation processes (Cadle and Williams, 1980; Unice et al., 2019b). While some work has given mechanical insights that rubbers such as polystyrene-butadiene and poly-isoprene undergo photodegradation (Wypych, 2015), the studies of Cadle and Williams remains the only one to provide a degradation rate. Recent lab-scale investigations into the effects of photodegradation also do not report degradation rates as these have focussed on the effects on leaching and transformation of tyre additives (Thomas et al., 2022; Unice et al., 2015).

The degradation rates used in the currently available modelling studies are mostly based on the research of Cadle and Williams from 1980 (Cadle and Williams, 1980; Unice et al., 2019a). As Corella-Puertas et al. mention, this is the only experimental study to provide a half-life of tyre wear particles in soil and there are no studies on the degradation of TRWP in the aquatic environment (Corella-Puertas et al., 2022). As the composition of tyres has changed over the 40 years since the study, the results of Cadle and Williams may be less relevant now than when they were first published. Therefore, updated degradation rates for TRWP would result in a big improvement in the accuracy of modelling. Combining this with state of the art physicochemical characterisation of TRWPs as a result of ageing will further contribute towards understanding particle transport.

The aim of this research is to characterise the effect of UV-degradation on TRWP and provide updated degradation rates for TRWPs. To achieve this, samples have been aged following an accelerated UV-ageing procedure. To test the degradation of pure tyre tread, tyres have been cryomilled, which is recognized as an efficient method to reduce the particle size of rubber (Thomas et al., 2022; Bar-Cohen and Radebaugh, 2016). It has also been reported that the wear process that generates TRWPs under real conditions also causes significant chemical changes in the tread rubber, with studies showing that more rubber is unvulcanised so the material absorbs oxygen much faster than tread rubber which has been cryomilled to the same particle size (Cadle and Williams, 1980). To investigate the effects of realistic wear, we use simulated TRWP generated at a road simulator. Thermogravimetric analysis (TGA) is used to investigate the degradation rate whilst static light scattering (SLS), electron microscopy (SEM-EDX) and gas chromatography (GC-MS) are used to characterise the physicochemical changes of the particles due to UV-ageing.

2. Materials and methods

2.1. Materials

Tween-20 (Sigma, Lot # SLCC6187) was used as received without further purification. All water used was MilliQ filtered using a Millipore 0.22 μm filter. Five different models of light duty tyres were used representing summer, all-weather and winter types and premium and budget market segments. These were purchased from BandenConcurrent. Shredded end-of-life heavy duty tyres were provided by Rumal Tyre Recycling.

2.2. Tyre wear particle generation through cryomilling

To produce model TWPs, small sections of tyre tread were removed from the running surface of four different models of brand new premium light duty tyres using cutting pliers. The shredded end-of-life truck tyres were used as received and may contain both running surface and sidewall rubber. These rubber chunks were then milled in a Ruhromag Retsch ZM-1 centrifugal mill with a 250 μm screen. Milling was performed under LN_2.

2.3. Tyre and road wear particle generation

TRWPs were generated at VTI's road testing facility using the same four premium brand models as the TWPs and one budget brand light duty tyre (Gustafsson et al., 2024). Particle sampling in the simulator hall makes it possible to sample TRWPs with very low contamination from surrounding sources and no influence from tail-pipe emissions. The road simulator consists of four wheels that run along a circular track with a diameter of 5.3 m. The axle load in the simulator was 550 kg per axle and tyre inflation pressure was 4 bar, which compares to larger cars (i.e. SUVs) weighing around 2200 kg. The speed of the road simulator was 70 km/h, which is the maximum possible speed. A cement concrete pavement ring was used for the tests, consisting of rock ballast with a maximum aggregate size fraction of 11–16 mm. The friction coefficient of the pavement, measured with a portable friction tester was between 0.72 and 0.85 and the macro texture, measured with a line laser texture meter, presented as mean profile depth was between 0.55 and 0.95 mm. These numbers indicate a lower surface roughness than asphalt. A photograph of the simulator is available in the Supplementary Information, Fig. S1. During the test time the simulator hall was not ventilated although pressure gradients might have caused minor self-ventilation. An internal air-cooling system was used to temperate the simulator hall. Sampling of coarse tyre wear fractions were made using 1 × 1 m aluminium deposition plates placed on the floor. These were vacuumed after each test using a Dyson cyclone vacuum cleaner. The collected material was stored in a standard freezer at −18 °C.

2.4. Fractionation

Sieving of cryomilled TWP and TRWP was performed in MilliQ water with a drop of Tween-20 using stainless steel sieves of 53 and 200 μm. The fraction between 53 and 200 μm was collected and dried at 60 °C.

2.5. Accelerated UV-Ageing

The accelerated UV degradation was performed in an Atlas Suntester XXL, with three Xenon lamps (air cooled), a light intensity of 60 W/m^2 between 300 and 400 nm, relative humidity of 45% and a chamber temperature of 60 °C and a black standard temperature of 81 °C. The samples were sprayed every 12 h for 1 min with demi-water to thus mimic outdoor conditions. This sprayed water evaporated within 1 h. Samples were taken after 160, 505 and 1000 h of ageing. As ageing was performed in petri dishes the water could not fully flow away from the particles so the extent to which leachates were removed from the particles is unknown.

2.6. Static light scattering (SLS)

SLS was performed using a Horiba LA-960S2 in a 10 ml fraction cell equipped with a magnetic stirrer to ensure particles maintain a well-dispersed suspension. Samples were measured in a 0.05% Tween-20 in MilliQ solution to reduce agglomeration and particle-cell interactions. Particle size distributions were calculated from the scattering results using a refractive index of 2.94–0.00I (Gustafsson et al., 2019; Järlskog et al., 2020).

2.7. Scanning electron microscopy with energy dispersive X-ray spectroscopy (SEM-EDX)

SEM-EDX analyses were performed with a Tescan MAIA III Triglav field emission scanning electron microscope, equipped with Bruker XFlash Quantax 30 mm^2 silicon drift detectors for energy dispersive X-ray spectroscopy. SEM images were recorded in the secondary electron (SE) and backscattered electron (BSE) modes between 5 and 15 kV.

2.8. Thermogravimetric analysis (TGA)

TGA was performed using a Mettler Toledo TGA 2 system following the method used by Son and Choi (2022). The experiments were performed under nitrogen with a heating rate of 20 °C/min from 40 °C up to 750 °C before switching to air and continuing heating until 1000 °C. The mass loss with time and temperature is monitored.

2.9. Direct thermal desorption gas chromatography mass spectrometry (DTD-GCMS)

DTD-GCMS on semi-volatile organic compounds was performed with an automatic thermal desorption unit (ATD Turbomatrix 400, PerkinElmer) coupled with a capillary gas chromatograph (HP 6890, Hewlett-Packard) and a mass spectrometer as detector (Agilent 5973, Agilent technologies). A metal tube was filled with 1–2 mg subsamples of the pristine and UV aged T(R)WP in the petri dishes, without further sample treatment. The tube was heated to 340 °C under a helium flow for 15 min to desorb and release the SVOCs from the T(R)WP matrix. The desorbed components are captured on a cold trap and injected onto a GC-column (VF624MS, 30 m × 0.25 mm i.d. 1.4 μm film thickness; Agilent) via flash heating. Identification was achieved via comparison of retention indices and mass spectra based on the TNO-AMDIS and NIST libraries. Quantification is performed with an external standard mixture. For compounds without a calibration standard, the content is calculated semi-quantitatively based on the response of toluene and naphthalene. Individual SVOC compounds are classified by their functional groups.

3. Results and discussion

In this research, four T(R)WP samples were used and these are summarised in Table 1. The choice of the acronyms TWP and TRWP was based upon the naming conventions proposed by Halle et al. in attempt to ensure harmonisation in terminology between studies (Halle et al.,

Table 1
Summary of T(R)WPs samples used in this study.

Sample	Tyre Details	Rubber composition [a]		Filler [a]	Particle generation
		NR	SBR + BR		
Premium Car TWPs	Mix of four new premium segment tyres (2 brands: 2x summer, 1x all season, 1x winter)	20%	80%	SiO_2	Cryomilling
Truck TWPs	Mix of shredded end-of-life truck tyres from tyre recycler	80%	20%	carbon black	Cryomilling
Premium Car TRWPs	Mix of four new premium segment tyres (2 brands:, 2x summer, 1x all season, 1x winter)	20%	80%	SiO_2	Circular Road Simulator
Budget Car TRWPs	One new budget segment summer tyre	35%	65%	carbon black	Circular Road Simulator

[a] Determined with TGA and SEM-EDX.

2020). Two samples of tyre wear particles (TWPs) were prepared by cryogenic milling to investigate the effects of UV degradation purely on tyre material. A mix of light duty (car/van) tyres and heavy duty (truck) tyres were compared as the former generally contains more synthetic rubber and the latter more natural rubber, as reported in the ISO specification for TRWP analysis through pyrolysis GC-MS where the assumption is that car tyres contain 44% SBR + BR whilst truck tyres contain 45% NR (ISO, 2017; "ISO, 2017). The other two samples were prepared by running tyres on a circular road simulator with a concrete cement pavement at VTI's tyre testing facility and collecting the TRWPs generated. This allows for investigation of more realistic particles that also include road wear encrustations. TRWPs were prepared from the same mix of light duty tyres, which were from the premium market segment, as the cryomilled car TWPs in order to investigate the influence of road wear encrustations and the wear process. TRWPs were also collected from a budget segment light-duty tyre, which contains different fillers (i.e. carbon black) and rubber formulations. Finally, all samples were sieved to a size fraction of 53–200 μm to be representative of T(R)WPs that are deposited in roadside soils, the environmental compartment with the most T(R)WPs.

T(R)WPs were subjected to accelerated abiotic ageing with samples taken for characterisation at four time points (0, 160, 505 and 1000 h). These time steps were taken pragmatically at around 1, 3 and 6 weeks. When converting the accelerated exposure in the UV cabinet to environmentally relevant timescales, the intensity of the UV light and the temperature need to be compared with natural conditions. Since we have only done UV ageing experiments at one temperature and one UV intensity, two assumptions were made: that thermal activation increases by a factor of 2 for every 10 °C and that UV-activation increases linearly. The first assumption is taken from several references on rubber ageing, where activation energies at room temperature degradation of 45–50 kJ/mol were reported, leading to an acceleration factor of 1.8–2 (Bauer et al., 2007; Pazur and Petrov, 2015). The second assumption on the linearity of UV degradation is taken from the papers from Therias et al. and Martin et al. (Martin et al., 2003; Therias et al., 2021), where many polymers show an almost linear dependency on UV intensity. In addition, the intensity of the UV light used (60 W/m^2 at 300–400 nm) is almost similar to the reference equatorial solar intensity that was presented in various CIE and ISO standards (How Sun Came Into Standards). This means that the acceleration factor approaches 1 anyway. Therefore, the error caused by the linear assumption will be small.

This reasoning leads to a total acceleration factor of 24 (16 for temperature difference between 60 and 20 °C, and 1.5 for the UV intensity difference between 60 and 40 W/m^2 at 300–400 nm), with an estimated error of 4. Given 1000 h of accelerated ageing and assuming an average sunshine exposure of 8 h per day in the environment, this leads to a simulated environmental ageing time of 8.2 years. However, this average sunlight exposure can be significantly lower, when TRWP are embedded in soil or water. Then the UV light that these particles are exposed to can reduce from 40 W/m^2 to <10 W/m^2 (at 300–400 nm). This will increase the corresponding exposure time to ~32 year. In conclusion, the environmental exposure time of 8 ± 2 years is valid for TRWP on the road, and 32 ± 8 years for embedded particles in soil or water. These values have been used for calculating abiotic environmental degradation rates.

3.1. Degradation rates of T(R)WPs

Thermogravimetric Analysis (TGA) was used to investigate the degradation rate of T(R)WPs. As different components of T(R)WP will be lost at specific temperatures it is possible to determine the composition of the different samples and how this changes with ageing.

The composition of the four samples as determined by TGA is shown in Table 2. The composition is as expected for standard tyres, namely 40–60% rubber, 20–30% filler (SiO_2 or carbon black (CB)) and 12–15% oils (Knight et al., 2020). The car TWPs prepared by cryomilling have a much higher inert fraction compared to Truck TWPs. This is due to the inclusion of SiO_2 as a mineral filler in premium car tyres, whereas the main filler for truck tyres is CB. The two TRWP samples have much higher inert fractions due to the presence of road wear particles which are mainly aluminium and silicon-rich minerals.

Table 2
Composition of T(R)WP samples as determined by TGA.

	Semi-volatiles	Rubber	Carbon Black	Inert
Car TWPs	6.5	52.8	4.1	36.6
Truck TWPs	4.6	60.8	29.0	5.6
Premium TRWPs	1.6	10.1	6.5	81.9
Budget TRWPs	1.5	7.6	5.7	85.2

Fig. 1A shows the TGA trace of the car tyre sample in black (upper curve in plot) with the different components annotated on the right-hand side (plots for the other samples are available in the Supplementary Information, Fig. S2). There is a gradual weight loss until ~290 °C, which is due to the evaporation of semi-volatile organics present such as extender oils, softeners, vulcanisation agents and antioxidants. From 290 to 550 °C there is a sharp weight loss event due to pyrolysis of tyre rubber. At 750 °C, the nitrogen environment is changed to air and this leads to a weight loss event at ~800 °C due to the oxidation of carbon black. The remaining weight is referred to as inert material or ash and for the TWP samples this is mainly due to SiO_2 filler. In the TRWP samples, road wear encrustations, consisting of oxides of silicon, aluminium and calcium, contribute to the amount of inert material. The amount of mineral material is higher than is expected from literature (Unice et al., 2012; Kreider et al., 2010), which assumes 50% road encrustations in TRWPs. If we start from the same assumption, the amount of TRWPs in the total TRWP sample is around 40%, the remaining mass being unattached road wear particles from the cement concrete pavement.

The tyre wear to road wear ratio of the road simulation tests is different to the ratio between on-road emission estimates of tyre wear (100 mg/vkm (Giechaskiel et al., 2024b)) and road surface wear (140 mg/vkm (Deltares, 2016)). As the total tyre abrasion rate with the road simulator (20–45 mg/vkm) was 2–5 times lower compared to the tyre wear emission estimates of 100 mg/vkm, the road simulator approximates the emission of road surface wear but underrepresents the emission of tyre wear. Although these findings indicate that it remains a challenge to mimic real world conditions with a road simulator, SEM-EDX observations show that particle properties (shape, morphology, road encrustations) of the generated TRWPs are similar to real-world TRWPs (Kreider et al., 2010). However, when interpreting the degradation results, the large amount of road wear that may block UV light must be taken into account.

The red line (lower curve) in Fig. 1A shows the differential of the TGA trace (DTG) curve (black, upper line). Using the differential, small changes in composition are elucidated. For example, the weight loss between 290 and 550 °C due to rubber is shown to actually contain two events centred ~390 °C and ~480 °C. Previous work has shown that different types of rubber pyrolyse at different temperatures and these two peaks correspond to natural rubber (NR) and synthetic rubber (SBR + BR), respectively (Sun et al., 2022). This is also confirmed by analysis of rubber standards and is presented in the Supporting Information, Fig. S3.

The rubber degradation of the four different samples is shown Fig. 1B. All samples seem to follow a first order decay. The cryomilled car tyres and truck tyres show very similar accelerated degradation rates of 0.098 and 0.095 h^{-1}, respectively. When corrected for the acceleration factor this results in abiotic environmental degradation rates of 0.034 (0.027–0.045) and 0.032 (0.026–0.043) day^{-1} for exposed particles on the road and 0.0084 (0.0067–0.011) and 0.0081

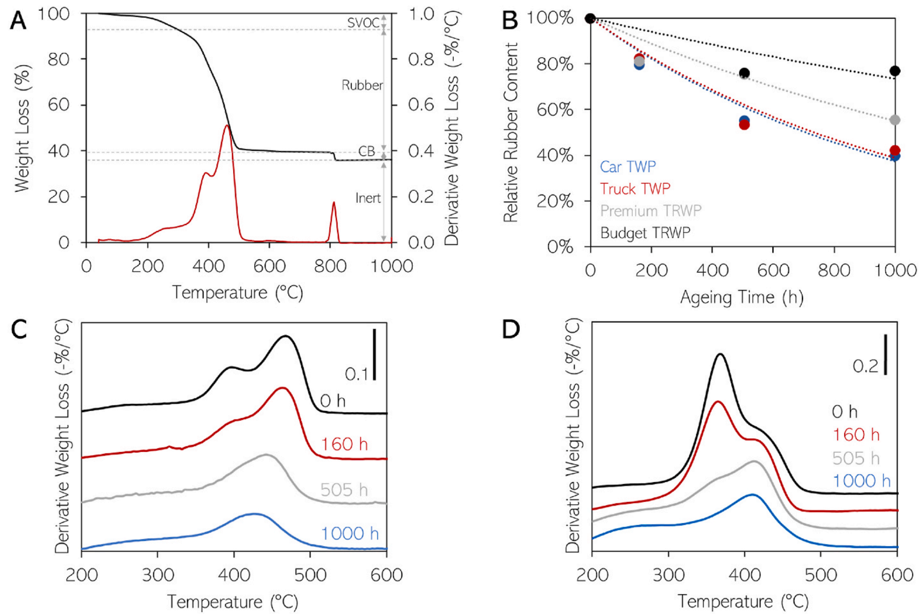

Fig. 1. **A.** TGA (black) and DTG (red) plots of the Car TWPs sample before ageing; **B.** Rubber concentration of the four T(R)WP samples as a function of ageing time; **C.** DTG peaks of rubber for the Car TWPs sample at the four ageing times; and **D.** DTG peaks of rubber for the Truck TWPs sample at the four ageing times.

(0.0065–0.011) day^{-1} for particles embedded in soil. The samples from the road simulator had a much slower degradation rate. This is contrary to previous work that suggests that particles formed through wear absorb oxygen faster and may be due to a shielding effect of road wear encrustations blocking the rubber from UV exposure. The premium TRWPs, which are the same tyres as the car TWP sample but run on the road simulator, had an abiotic environmental degradation rate of 0.020 (0.016–0.027) day^{-1} for particles on the road and 0.0051 (0.0041–0.0068) day^{-1} for particles embedded in soil. Finally, the budget TRWPs show the slowest degradation with rates of 0.014 (0.011–0.019) day^{-1} and 0.0036 (0.0029–0.0048) day^{-1} for the road and soil scenarios respectively. Cadle and Williams studied unaccelerated environmental degradation by exposing TWPs and TRWPs in soil and glass beads for a period of 16 months. The TWPs and TRWPs in glass beads are assumed to be representative of unaccelerated abiotic degradation and may be compared with the Car TWP and two TRWPs in this study. The TWP in the previous study showed no degradation, whilst in this study they show the fastest. TRWPs in glass showed a degradation rate of 0.09 day^{-1} which is approximately 5–10x faster than the TRWP samples in this study. Cadle and Williams also showed that degradation in soil was roughly twice as fast (0.15 day^{-1}) highlighting the important role biodegradation plays in the environmental fate of T(R)WPs. Moreover, in this study we determined degradation rates using 53–200 μm particles whilst the environmental particle size distribution is bimodel with a peak between 5–25 μm and 50–200 μm. As degradation processes occur mainly at the surface, smaller particles with a larger surface degrade faster, which may also lead to higher degradation rates for TRWPs in the environment.

The degradation rate is likely influenced by the type of rubber present in the tyres. As mentioned previously, using the DTG plot we can identify both natural and synthetic rubber. In Fig. 1C, the rubber region of the DTG plot for cryomilled car tyres is shown with increasing ageing time. The fresh sample shows two peaks, *ca.* 390 °C and *ca.* 480 °C which have been shown to be due to NR and SBR + BR, respectively (Sun et al., 2022). It is known that the two types of rubber degrade at different rates and this is also observed here, with the peak due to NR becoming indistinguishable after 500 h ageing (Baensch-Baltruschat et al., 2020b). This was also confirmed using TED-GCMS for quantification of NR and SBR + BR as shown in Table S3 in the Supplementary Information. There also appears to be a shift of the synthetic rubber peak to lower temperatures with increased ageing, with the peak being centred around 430 °C after 1000 h ageing. This may be due to a chemical change in rubber occurring during ageing, such as devulcanization, photooxidation or thermo-oxidation, that lowers the degradation temperature of the rubber. The aged truck tyres also show two peaks, as shown in Fig. 1D, this time *ca.* 390 °C and *ca.* 435 °C. The shifted degradation temperature of the synthetic rubber with respect to that in the car tyres may be due to a different type or ratio of synthetic rubber(s) or, as these are end-of-life tyres, a different degree of vulcanisation. It is evident from the DTG that truck tyres contain a much larger proportion of natural rubber than synthetic rubber and this is again seen to degrade much quicker than the synthetic rubber. Direct comparison between the truck and car tyres is difficult as the start material differs in state and composition: one is end-of-life so has been subjected to years of external forces whereas the other is unused, the end-of-life sample may include sections of sidewall whereas the car tyre contains only tread from the running surface. However, in both samples we see that the NR component degrades quicker than SBR + BR so it can be assumed that in the environment natural rubber truck TRWPs degrade quicker than car TRWPs.

3.2. Effect of ageing on organic additives

To investigate the transformation of organic compounds by UV ageing, virgin and 1000 h UV aged car TWP and truck TWP were analysed with DTD-GCMS for non-target screening of semi-volatile organic compounds (SVOCs). In Fig. 2 results of the SVOC screening are shown, where SVOC compounds are grouped by their chemical class (functional groups) and presented as summations of the compound total ion chromatogram (TIC) peak areas. By 1000h UV ageing the total amount of SVOCs is reduced to respectively 40% and 50% of the initial SVOC amount in car TWP and truck TWP. This is mainly caused by the decrease in paraffinic, olefinic and aromatic compounds (extender oils), benzothiazoles (vulcanisation accelerator) and amines (anti-oxidants). In contrast, there is an increase in oxygenated compounds (carboxylic acids, aldehydes, ketones and phenols). This is in agreement with the UV-induced oxidation pattern, from non-oxygenated species, via alcohols, carbonyls to carboxylic acids. These results indicate that UV aging results in partial oxidation of the organic additives in TWP. However, it's still unclear what are the main removal paths of these organic

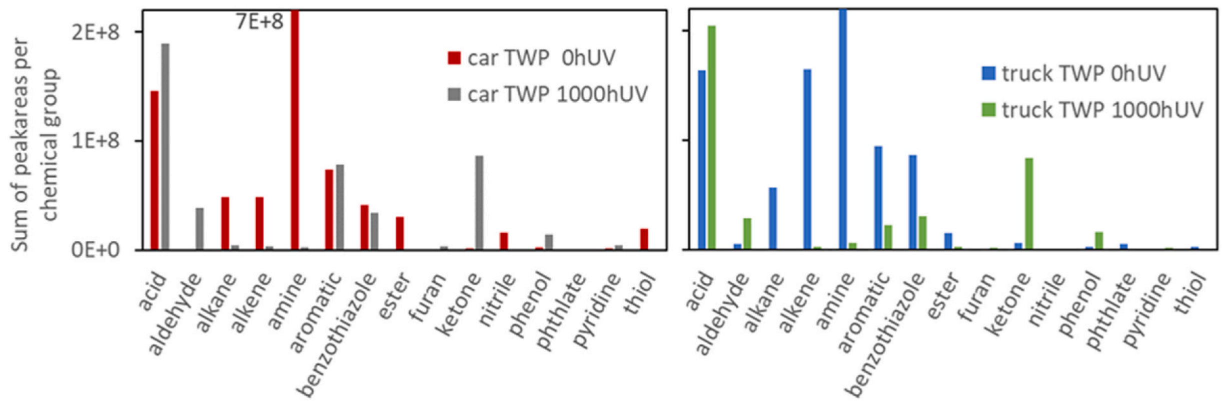

Fig. 2. Results of the DTD-GCMS screening of semi-volatile organic compounds in car TWP (virgin and 1000h ageing) and truck TWP (virgin and 1000h ageing). Compounds are grouped by their chemical class and presented as summations of the compound total ion chromatogram (TIC) peak areas.

compounds: volatilisation (either direct or indirect after UV-induced breakdown into more volatile compounds) or leaching (either direct or indirect after UV-induced oxidation into more water soluble compounds) among others.

3.3. Effect of ageing on T(R)WP size

It is also important to understand what influence degradation has on the physicochemical properties of T(R)WPs. This is of importance as a change in particle size will lead to different environmental transport behaviour, such as sedimentation rate in water and (re)suspension in air affecting long-range transport. It is also expected that smaller particles will be taken up more easily by biota and present a potentially higher health effect. Static light scattering (SLS) was used to investigate changes in particle size with ageing and these are shown in Fig. 3.

The modal particle size of the virgin car tyre sample (Fig. 2A) lies around 200 μm which is representative of the coarser TRWPs found in roadside soils. When looking at the samples that have been aged, the modal size of the TWPs clearly shifts to lower particle size with longer UV-exposure. From this we can calculate a particle size reduction rate of 0.03 (0.02–0.05) μm day^{-1}. A peak is also seen emerging around 10 μm and this peak intensifies as the sample has been aged longer. This bimodal distribution suggests that both surface degradation and fragmentation play a role.

The modal particle size of the truck tyre sample (B) is similar to the one of the car tyre sample, around 175 μm. However, the virgin sample also seems to have a part of the fraction with particle size larger than 1 mm. This could indicate the formation of agglomerates in the sample, even though a surfactant was added before analysis to prevent this. The "stickiness" of natural rubber containing truck tyres has previously been

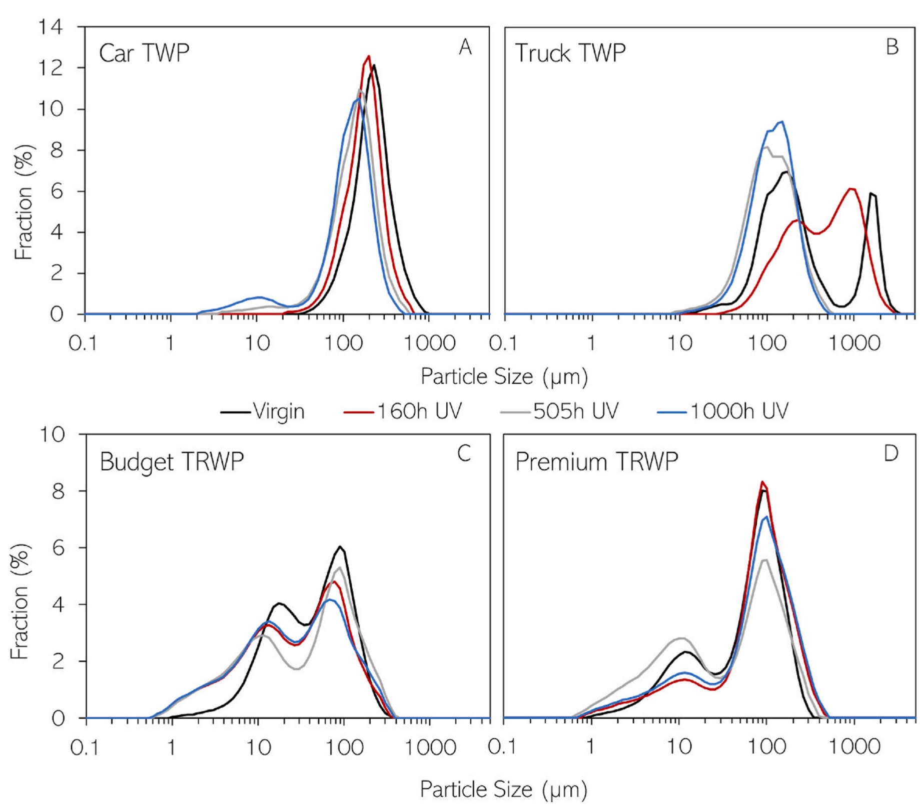

Fig. 3. Volume distribution measured with Static Light Scattering (SLS) from **A.** Car TWP, **B.** Truck TWP, **C.** Budget TRWP and **D.** Premium TRWP. Each graph shows four lines that correspond to different ageing times: virgin (black), 160 h (red), 505 h (grey) and 1000 h (blue).

reported as a limiting factor in the particle size analysis of truck tyre cryogrinds (Thomas et al., 2022). The sample that has been aged for 160 h also shows a bimodal distribution, however, the 505 and 1000 h samples have a monomodal distribution. This is in line with the differential thermogravimetry (DTG) data presented earlier which showed that NR was almost completely degraded after 505 h ageing and the sample is predominantly synthetic rubber. It could also be that the surface of the particles changes such that agglomerates are less likely to form after ageing by UV-light, for example through formation of carbonyl groups increasing hydrophilicity. Furthermore, after ageing more than 500 h, the tail of the peak starts to shift to lower particle sizes, which indicates the formation of more particles around 10 μm.

A difference between the milled TWP distributions and the budget (C) and premium (D) TRWP distributions, is the bimodal distribution of the unaged TRWP. Since the smaller particles are not present in the TWP samples, this could represent loose road wear in the sample. Whilst the samples were fractionated to between 53 and 200 μm during preparation, ultrasonic treatment during dispersion for SLS measurement could liberate loosely bound road wear encrustations from the TRWPs. The budget TRWP sample (C) shows a shift of the distribution towards smaller particle sizes. The fraction of particles *ca.* 100 μm is decreasing with longer degradation times, whilst the fraction of particles smaller than 10 μm is increasing. Different from the milled tyre samples, nanoparticles <1 μm are also formed with longer UV exposure time. This increase in particles <1 μm is also clearly visible in the premium TRWP sample (D), however, these distributions do not show a clear shift like the other samples.

3.4. Morphology and elemental composition of (aged) T(R)WPs

In the SLS results we see in many samples the size fraction <10 μm growing. For the car tyre sample we can say with certainty that these particles originate from tyre material, however for the TRWP samples it is not possible to say whether this fraction is due to rubber, road wear, or both. With SEM-EDX we can investigate both the morphology and elemental composition of individual particles, which may shed more light on this important smaller fraction.

In Fig. 4A particles from <10 μm in the 1000 h aged car TWP sample are shown, together with their EDX spectrum in Fig. 4B. The SEM analysis confirms the SLS results that there are a considerable number of particles <10 μm present in the sample. EDX analysis shows that these particles consist of a mixture of carbon (C), oxygen (O) and silicon (Si). The two Au peaks present are due to the gold-coated filters used to prepare the samples.

The SLS results show that the TRWP samples from the road simulator also contain particles <10 μm which is again confirmed by SEM analysis.

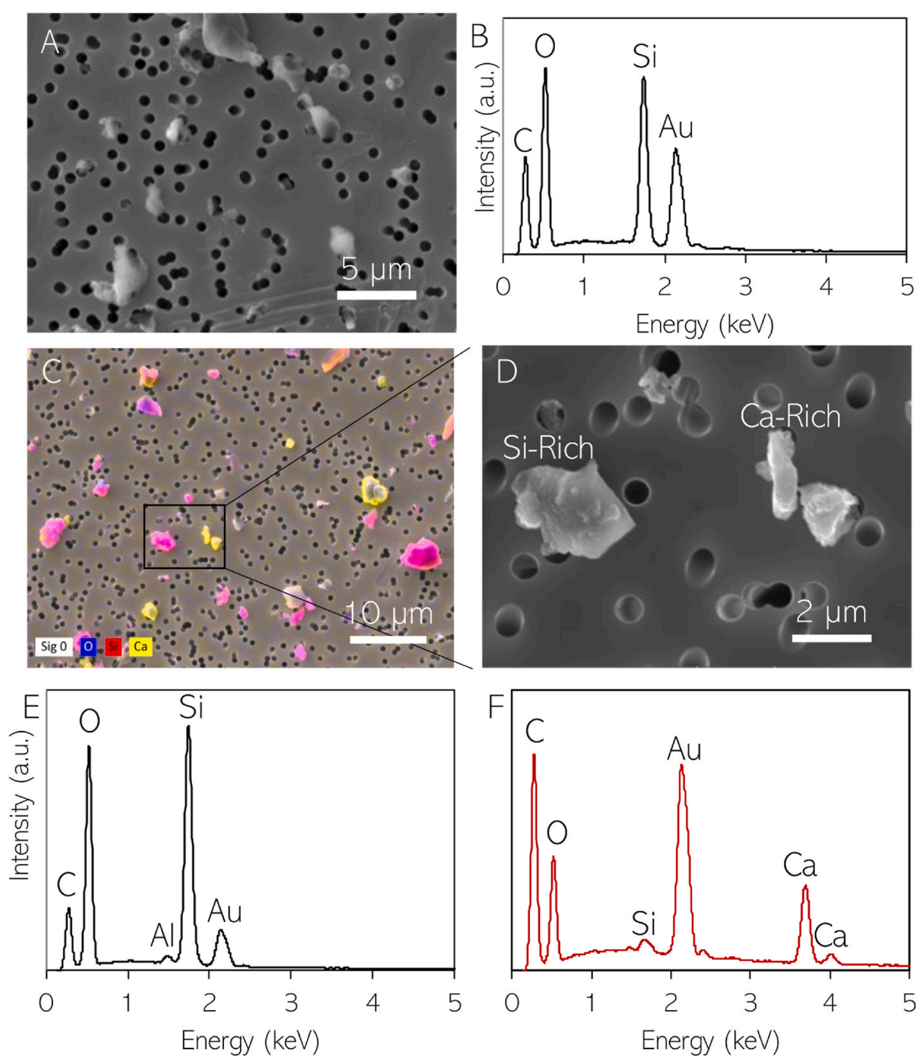

Fig. 4. **A.** SEM image of particles <10 μm after 1000 h ageing of car TWPs; **B.** EDX spectrum of small car TWP particles. **C.** Elemental mapping of particles <10 μm in the 1000 h aged Premium TRWP sample; **D.** SEM image of suspected tyre wear and road wear particles indicated by the box in A; **E.** EDX spectrum of the Si-rich particle; **F.** EDX spectrum of the Ca-rich particle.

However, not all of these particles have the same elemental composition. Fig. 4C presents an elemental mapping of the particles in the premium TRWP sample after 1000 h ageing and it can be seen that this sample contains at least two types of particles <10 μm: calcium (Ca) rich particles (yellow) and silicon (Si) rich (pink). A higher magnification image of a couple of these particles is show in Fig. 4D. Here, the Si-rich particle shows a similar morphology to the particles shown in Fig. 4A, whereas the Ca-rich particles have a slightly rougher edge. EDX spectra of these particles are shown in Fig. 4E + F and here we also see that the Si-rich particle has a similar elemental composition to the aged TWPs presented in Fig. 4B, namely C, O and Si. However, both the tyres and the road surface used in this experiment contain silicon and either could be the source of these particles. The presence of high concentrations of calcium (Ca) and small amounts of aluminium (Al) originates from the road surface of the simulator which uses a cement running track, the composition of which can be found in Table S4 in the Supplementary Information.

To get more insight into the origin of these particles <10 μm, the budget TRWP sample was also analysed with SEM-EDX. These tyres do not contain SiO_2 filler (<0.5%) and the tyre wear mainly consists of C with small amounts of O, zinc (Zn, ~2%) and sulphur (S, ~1.5%). Therefore, it would be expected that TRWP originating from these tyres have less silicon than the premium TRWP making it easier to distinguish the tyre material from the road wear particles. Fig. 5A, B, D and E show that larger budget TRWPs, virgin as well as 1000h UV aged, indeed contain very low concentrations of Si. The smaller particles in the 1000 h UV aged budget TRWP sample, shown in Fig. 5C and F, contained higher concentrations of Si than the large particles and a higher concentration of C than the Premium T(R)WPs shown in Fig. 4. As the budget tyres contain little to no Si, this would indicate that all analysed particles <10 μm in the budget TRWP sample are either road wear or a combination of tyre and road wear and none of the observed particles are only tyre wear. This is remarkable as the SLS graph (Fig. 3C) shows that particles <10 μm become continually smaller with longer UV exposure. Since road wear is mainly inorganic and should not be influenced by the UV, it could be that smaller (inorganic) particles are released from the bigger TRWP. From the car TWP sample (Fig. 4A + B) we can say that the small particles formed are definitely tyre wear, as the only type of particle in this sample is tyre. This means that most likely small TWP are also formed in the TRWP samples, however in these samples it is a challenge to distinguish small tyre wear particles from small road wear particles as the degradation leads to a loss of characteristic TRWP elements (C, S, Zn) and the vast majority of particles in the sample are road wear.

Next to the analysis of particles <10 μm, SEM can also give an insight into the morphology and composition of the larger particles. When using the back scattered electron (BSE) detector, information on the composition of the particles is gained, with heavier elements appearing brighter in the image. Fig. 6A+B show representative examples of a virgin car TWP and a particle that has been aged for 1000 h, respectively. Looking at the morphology of the particles, it is noticeable that the aged particle is smoother than the virgin particle, further suggesting that fragmentation plays an important role in the degradation process. This is also the case for the truck tyre sample (Fig. S5 in the Supplementary Information). When comparing the particles there is a clear difference in the colour of the particles in the BSE images. The virgin particle is darker which means it contains more lighter atoms than the aged particle. This is also observed in the EDX spectra of the two particles, shown in Fig. 6C. The virgin particle contains more carbon (C) than the aged particle, whereas the concentration of oxygen (O) and silicon (Si) are much higher in the degraded particle. The inset of Fig. 6C also shows the area of the spectrum with S. S is present in low concentrations due to its use as a vulcanising agent and it remains present in cross-linking bonds of vulcanised rubber. The S seems to be present at much lower concentrations, if at all, in the 1000 h aged sample. This could suggest that UV ageing degrades cross-linkages in the rubber leading to devulcanization. This could explain the observations in Fig. 1D where a shift in the decomposition temperature of synthetic rubber is observed. The C:Si ratio of fresh and 1000 h aged samples is further explored in Fig. 6D.

3.5. Indications of degradation in the environment

We also see clear indications of degradation in the environment. Field measurements were performed at roadside locations in the Netherlands (i.e. highway A2, city intersection Rotterdam and provincial road N201) with samples of road runoff, atmospheric deposition and soil. All samples were analysed with TED-GCMS to determine the concentration of TWP. Details on the measurement campaign, the methods

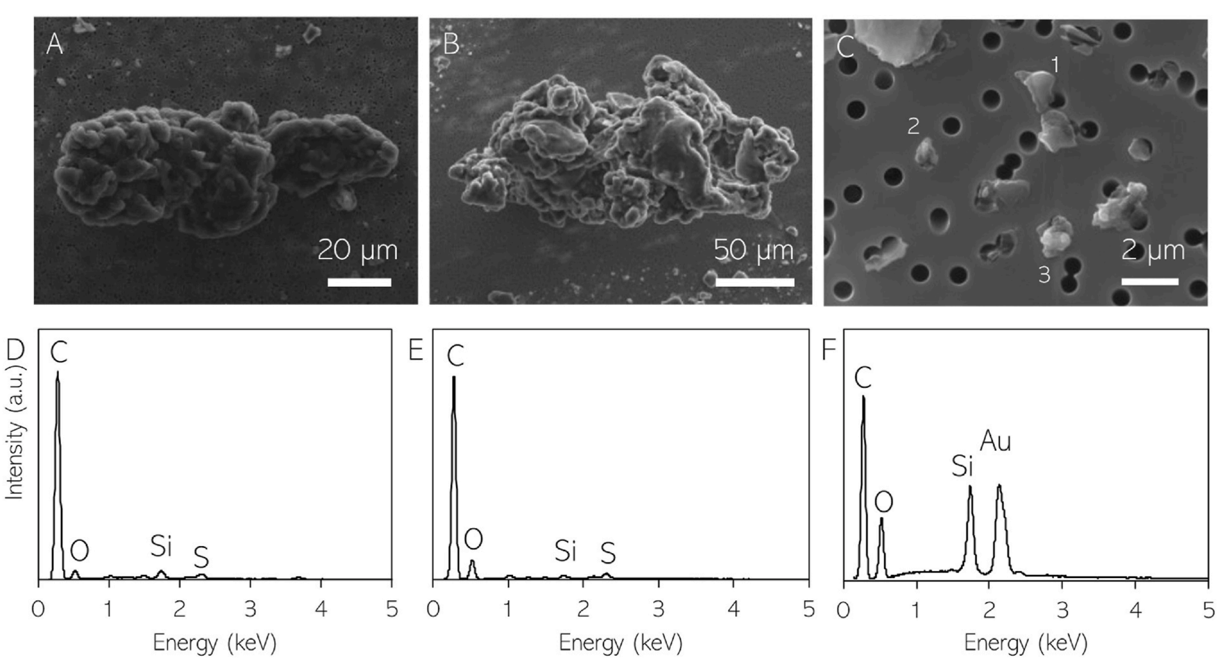

Fig. 5. SEM images and EDX spectra of large virgin (**A and D**) and 1000 h UV-aged (**B and E**), and small 1000 h UV-aged (**C and F**) budget TRWPs. The spectrum for the particle labelled 1 in C is shown in F and particles 2 and 3 can be found in the Supplementary Information, Fig. S4.

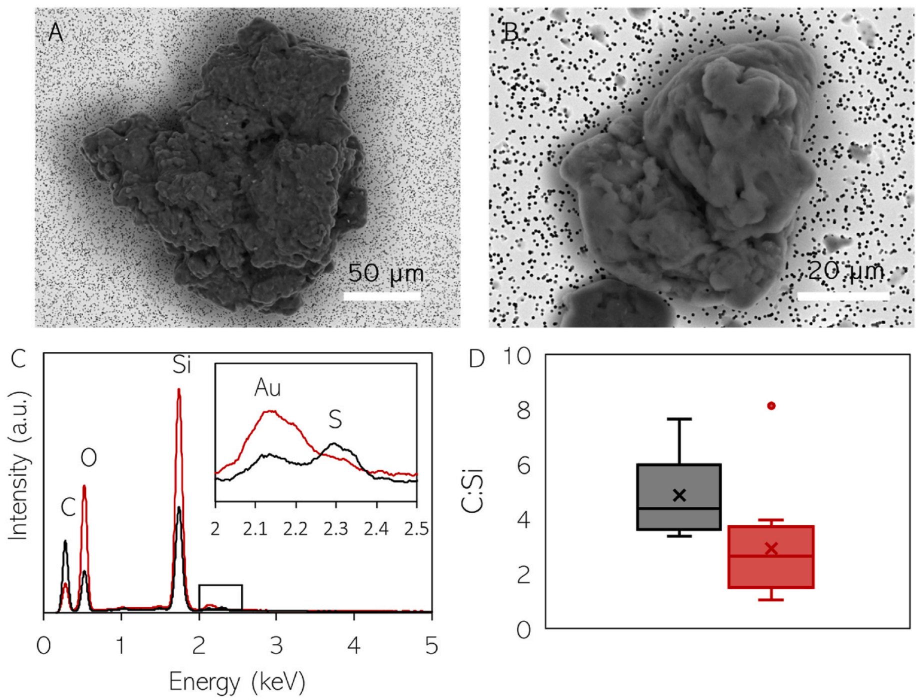

Fig. 6. **A.** BSE image of a virgin car TWP; **B.** BSE image of a car TWP after 1000 h ageing; **C.** EDX spectra of the virgin (black) and aged (red) car TWP particles; **D.** Box plot of C:Si ratio of virgin (black) and aged (red) car TWP particles determined using EDX of 10 particles from each sample.

used and results are available in the Supplementary Information. From the TWP concentrations in soil and in deposited dust the accumulation time of TWP in soil could be calculated. The accumulation time is calculated as the number of years of atmospheric deposition (in mg/m^2/day) taken to reach the measured soil concentration (in g/m^2) sampled on the same spot as the deposition samplers. Fig. 7 shows the accumulation times of TWP at the three roadside sampling locations in the Netherlands. In the same samples, copper (Cu) was also analysed with ICP-MS as a tracer for brake wear particles (BWP) (Hulskotte et al., 2014; Gietl et al., 2010). This makes it possible to compare the accumulation time of TWP (which is susceptible to UV and biodegradation) and BWP (consisting of metal alloys that do not degrade and leach). These accumulation times are based on a limited number of samples and as atmospheric deposition is greatly affected by meteorological conditions (i.e. wind direction, rainfall), the calculated deposition from the two monthly averaged samples have to be considered as 'snapshots' and can deviate from long term deposition values. This will also affect the calculated accumulation times and is the main reason for the variation between locations, depending on the orientation of the sampler relative to the road in relation to the wind direction. Because both BWP and TWP are analysed in the same samples, they are affected by the same meteorological conditions and therefore the quotient of TWP and BWP accumulation times gives an indication of degradation. Average accumulation times of TWP and BWP are *ca.* 40 years and 65 years, respectively, so the accumulation time of TWP is approximately 40% shorter than BWP suggesting a removal process at play for TWP but not for BWP, such as degradation.

We also see evidence of degradation when comparing the size distributions and the share of natural rubber of TRWPs in different environmental compartments. Fig. 8 shows the size distribution of TRWPs in samples of atmospheric deposition, road run-off and roadside soil. "Fresher" compartments such as deposited dust and run-off, where the particles haven't had long residence times and thus lower UV exposure, show more larger particles and a smaller percentage of particles <10 µm. In soil, a sink compartment with much longer residence times, the fraction of TRWPs <10 µm is higher. In combination with the size distribution results presented in Fig. 2, this could suggest that the extra

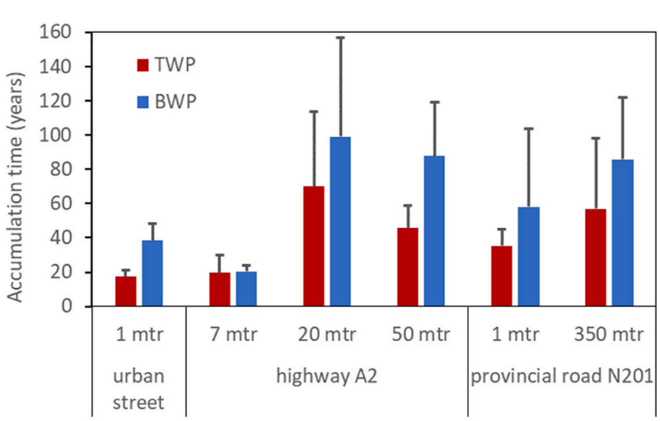

Fig. 7. The years of accumulation of TWP (red, n = 2) on several roadside soils in the Netherlands with different distances to the road compared to brake wear particles (BWP) (blue, n = 2).

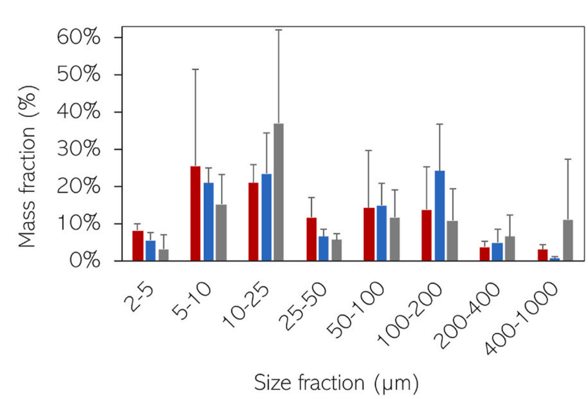

Fig. 8. Size distribution of TRWPs in atmospheric deposition dust (blue, n = 3), road run-off (grey, n = 3) and soil (red, n = 2).

particles <10 μm in soil could be attributed to particles generated through degradation. However, these are only initial observations and a more comprehensive study would make for important future research as there may be other factors that explain this observation. For example, road run-off may contain more large particles as it is closest to the source (tyre-road contact). Likewise, though soil and deposition samples were collected at similar distances from the road they were collected from different locations and this may also play a role.

When comparing the share of natural rubber in TWP in environmental compartments, we also see clear indications of degradation. At all three roadside locations in the Netherlands, the share of NR in TWPs in soil (12 ± 6%) is lower than the share of NR in TWP in deposited dust (22 ± 8%). This is in agreement with the observations from the accelerated UV ageing experiments. As could be observed in Fig. 1, NR degrades quicker than SBR + BR, so the share of NR decreases with longer residence times in the environment.

4. Conclusions

The fate of TWP in the environment is an understudied subject. This study takes the first steps towards an updated degradation factor for the abiotic degradation of TWP in the environment by quantifying polymer loss in accelerated ageing tests using cryomilled tyre tread and TRWPs from a road simulator. TGA analysis of the tyre composition as a function of ageing time showed that the average accelerated degradation rate was 0.074 h^{-1}, which when corrected for the acceleration factor leads to an abiotic environmental degradation rate of 0.025 (0.020–0.034) day^{-1} for exposed particles on the road surface and 0.0063 (0.0051–0.0084) day^{-1} for particles embedded in soil. As a relatively large size fraction was used, and approximately 50% of environmental TWPs are smaller than this, it could be that this is an underestimation due to the surface sensitive nature of degradation and the higher surface area of small particles. DTG analysis suggested that the different types of rubber within the sample degraded at different rates, with natural rubber degrading quicker than synthetic rubber. However, this was not reflected between samples with the heavy duty tyres showing the same degradation rate as light duty tyres. This may be due to the whole truck tyre being used and not only the tread layer. In addition, the degradation of TWPs is accompanied by a substantial loss of chemical compounds (mostly tyre additives) by UV-induced oxidation and breakdown.

Particle size also reduced during degradation with an environmental equivalent rate for the cryomilled car TWPs of 0.03 μm (0.02–0.05 μm) day^{-1}. A fraction of small particles <10 μm was also formed which was further investigated with SEM-EDX, where it was also observed that sulphur is removed during degradation. Finally, measurements in the field confirm the experimentally determined degradation mechanisms. Soil samples (with long term accumulation) contained a larger percentage of smaller particles when compared with atmospheric deposition samples which contain much “fresher” material. Future work should focus on further increasing the environmental relevance by using TWP that more accurately mimics that found in the environment, more precisely converting ageing times and investigating the effect of biodegradation.

CRediT authorship contribution statement

Marloes F. van Os: Writing – original draft, Visualization, Investigation, Data curation. **Merel G.A. Nooijens:** Writing – review & editing, Investigation. **Alex van Renesse van Duivenbode:** Writing – review & editing, Investigation. **Peter C. Tromp:** Writing – review & editing, Funding acquisition, Conceptualization. **Elena M. Höppener:** Writing – review & editing, Visualization. **Kalouda Grigoriadi:** Writing – review & editing, Investigation. **Arjen Boersma:** Writing – review & editing, Supervision, Funding acquisition, Conceptualization. **Luke A. Parker:** Writing – original draft, Visualization, Supervision, Conceptualization.

Consent for publication

All authors consent to publication.

Ethics approval and consent to participate

Not applicable

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Declaration of competing interest

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

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.chemosphere.2025.144121.

Data availability

Data will be made available on request.

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