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A novel method for assessing chemical leaching from surface water–pavement interactions applied to recycled-tyre reuse products

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Globally, recycling of otherwise waste materials into new products is desired. End-of-life tyres are increasingly incorporated into new pavement materials but leaching of entrained chemicals from such products is not well quantified. Chemical concentrations in runoff from pavements may pose environmental and human health risks. High liquid–solid ratio, batch-agitated leaching is standard practice for assessing leachability and hazards of chemicals-of-potential-concern in contaminated soil and wastes but is not reflective of important exposure scenarios and may mislead. A new static surface leaching procedure (SSLP) is introduced that is more representative of chemical leaching from pavement reuse materials whilst in contact with rainfall/runoff water. SSLP was evaluated over 2–14 d intervals against batch-agitated leaching for two rubberised pavement products containing 10-fold different proportions of crumbed end-of-life tyres. Although, batch leaching showed high mass removal of 1,3-diphenylguanidine (1,3-DPG, 34%) and hexamethoxymethylmelamine (HMMM, 30%), both batch- and SSLP-leached concentrations of 1,3-DPG, HMMM and N^1-(4-methylpentan-2-yl)-N^4-phenylbenzene-1,4-diamine quinone (6PPD-Q) were below ECOSAR-predicted toxicity thresholds for fish and daphnids. SSLP highlighted differences in chemical leachability based on rubber content of pavement products and offers a method applicable to other scenarios, such as PFAS leaching from concrete/asphalt pavements. The SSLP was shown to approximate one-dimensional leaching from the surface of the pavement and to be dominated by diffusive processes, thus yielding a simple repeatable approach.

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Environmental significance

Release of chemicals from waste reuse products may pose threats, despite global encouragement to reuse waste material. Methods are needed that represent reuse applications to best define risks. The new static surface leaching procedure (SSLP) represents chemicals leaching from pavement reuse materials whilst in contact with rainfall/runoff water. We tested SSLP for two rubberised pavement products containing 10-fold different proportions of crumbed end-of-life tyres; products with chemicals of potential concern such as 1,3-diphenylguanidine (1,3-DPG) hexamethoxymethylmelamine (HMMM), and N^1-(4-methylpentan-2-yl)-N^4-phenylbenzene-1,4-diamine quinone (6PPD-Q), shown to have toxic effects. The SSLP method provides actionable insights for improving material safety and regulatory practices.

1 Introduction

The circular economy has emerged as a vital framework for addressing global sustainability challenges by promoting the reuse, recycling, and recovery of materials that would otherwise become waste.[1,2] This approach is particularly relevant for high-volume waste streams such as end-of-life (EOL) tyres, which can be repurposed into valuable products like rubberised pavements, thus reducing landfill burdens and conserving resources. However, the successful integration of recycled materials into new applications requires more than just technical feasibility. It demands careful consideration of potential environmental risks, especially the possibility that hazardous chemicals may be transferred from the original waste to the new products.[3–5] As recycled tyre rubber is increasingly used in infrastructure, concerns about chemical leaching and subsequent environmental or health impacts underscore the need for cautious, science-based assessment.[6,7] This cautious approach is essential, as the presence of contaminants in recycled

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materials could undermine the very benefits the circular economy seeks to achieve.

Laboratory leaching methods are often used to classify the hazardous nature of contaminated soils, wastes and other materials such as concrete, to assess potential chemical release and the suitability of contaminated soils and waste for a range of purposes or landfilling.[8,9] Commonly used standard leaching tests include the United States Environmental Protection Agency (US EPA) Leaching Environmental Assessment Framework (LEAF)[10–13] and the Australian Standard Leaching Procedure (ASLP).[14] The US EPA LEAF consists of four methods (LEAF 1313 to 1316)[10–13] used to evaluate the leachability of chemicals of potential concern (CoPC) from a wide range of granular or monolithic materials. The ASLP is commonly used for contaminated soil or other hazardous waste classification and involves batch agitation of 20 : 1 liquid to solid ratio batches over 20 ± 2 h to assess the maximum leaching potential of CoPC from pulverised solids.[15] Rarely do such methods take account of the application domain or the environmental context for the sample being leached, where leaching may occur under ambient water contact conditions.

One such application domain, of increasing importance, is the potential for release and leaching of CoPC from paved surfaces. For example, where surfaces have become contaminated with hazardous chemicals such as per- and polyfluoroalkyl substances (PFAS) in concrete and asphalt[16–19] or where CoPC in waste materials such as end-of-life (EOL) tyres are embedded in reuse products such as roads, sidewalks and soft-fall materials.[2,20] Key to assessing risks to human health and the environment is quantifying total CoPC in contaminated pavements and waste materials and reuse products and determining the proportion of the total CoPC released under different exposure scenarios relevant to their application domains. In paved surface applications, water contact *via* rainfall or surface runoff is typically largely surficial, intermittent, low volumes and somewhat passive. As such, it is believed that batch agitation at high liquid–solid ratios, as in ASLP, may be less accurate in quantifying the leached concentrations of CoPC arising in such scenarios.

To better elucidate the leachability and bioavailability of chemicals, several leaching studies have been completed, targeting a range of complex materials and various CoPC. Intercomparison of leaching methods have been undertaken and advances in considering more passive, application-specific methods reported. None of the studies advanced pavement leaching methods. For example, recently, Navarro *et al.*[21] reviewed leaching methods with a focus on the potential release of PFAS. Rayner *et al.*[22] compared leaching methods on soils and established a less aggressive agitate and stand (static batch) method at a lower liquid-to-soil ratio to better mimic leaching from source zone soils through vadose zones to groundwater. They also compared the laboratory leaching method to *in situ* measurements of PFAS in pore water recovered from buried suction lysimeters in seeking to mimic more realistic (3 : 1) solid : water ratios.[23] Navarro *et al.*[21] used the methods to assess leachability from PFAS-stabilised soils. Leaching studies of construction materials are also documented within the literature but these have focused on the source materials to be used in construction or when waste materials are reused by cementation into a matrix[24] or for release from concrete or mortars.[25] Kosson *et al.*[26] specifically targeted waste and secondary materials but did not consider paved surfaces.

The focus of this study was on the evaluation of a novel static surface leaching procedure (SSLP) applicable to rubberised pavements where EOL tyre crumb has been reused. Using recycled waste materials, such as EOL tyre rubber in the production of rubberised asphalt, soft-fall surfaces used in children playgrounds and porous pavement materials exemplifies a pivotal strategy in advancing a circular economy, particularly given the widespread issues of tyre stockpiling, illegal dumping and landfill disposal.[2] However, the use of recycled tyre crumb rubber (RTCR) has not been evaluated extensively to understand the impact of CoPC on potentially sensitive receptors when RTCR is being reused in roads, pavements, and other secondary products. It is understood that some chemicals (*e.g.*, zinc and polycyclic aromatic hydrocarbons or PAHs) in tyres can potentially have negative environmental effects.[27] Similarly, tyre rubber anti-degradant/antioxidant compounds, such as N^1-(4-methylpentan-2-yl)-N^4-phenylbenzene-1,4-diamine (6PPD), hexamethoxymethylmelamine (HMMM), and 1,3-diphenyl guanidine (1,3-DPG) are added to tyres to stop premature degradation of the materials during use. These and associated transformation products such as 6PPD-quinone (6PPD-Q) are known to pose risks to aquatic life, notably impacting the health of fish species such as coho salmon, zebrafish, chinook and other aquatic organisms.[28–37] Outcomes from these studies highlight the need for rigorous environmental assessment and management practices to mitigate potential ecological risks associated with the use of RTCR in different applications, and to assess what is potentially leachable in reuse scenarios, compared, for example, to general use and stockpiling of tyres.

In the present study, we applied the SSLP to quantify the leaching of EOL RTCR that have been embedded in two pavement materials with RTCR mass fractions an order of magnitude different. The SSLP primarily reflects chemical leaching under conditions dominated by diffusion, without the influence of advective transport and without being fully immersed in water. The SSLP was adapted from the LEAF 1315 semi-dynamic tank leaching procedure, which, while rigorous, presents several practical limitations. Specifically, LEAF 1315 requires sealing all but one surface of the core monolith and installing a tubular (or another shape depending on the shape of the monolith) structure to confine water-surface contact, involving the use of sealants that may serve as unintended sorbents for target analytes. Furthermore, the extended sampling duration of 63 d in LEAF 1315 is not practical for routine laboratory operations. In contrast, the SSLP method would seek to employ shorter timepoints – here we evaluate five timepoints, ranging from 2 h to 14 d, which are designed to capture leaching behaviour over exposure periods relevant to typical surface water–pavement interactions, but primarily to align with LEAF 1315 testing intervals for comparative purposes.

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We applied this new static surface leaching procedure method to undertake leaching assessments that are reflective of pavement environments. Whilst the LEAF 1315 method accommodates monolith testing, pavements differ in exposing only an upper surface to water thus needing specific consideration. Similarly, while the ASLP method enables assessment under extreme conditions, such conditions are not always reflective of pavement environments. As indicated earlier, such 'surface leaching' testing is not only needed for tyre products but also for other paved surfaces that might release CoPC such as PFAS from intact concrete and asphalt pavements.[17,18] ASLP standard batch tests were run in parallel to compare results to the new SSLP method proposed here.

2 Materials and methods

2.1 Rubber products and their characteristics

Chemical compositional analysis and leaching investigations were undertaken on two pavement products (P1 and P2), both containing aggregate and RTCR. Both are used commercially for pavements but differ in the RTCR content. P2 has approximately a 10-fold greater mass fraction of RTCR than P1. Core samples of the manufactured products P1 and P2 used in the new SSLP testing were cylindrical with approximate dimensions of 5 cm length and 10 cm diameter (Fig. 1). A crumbed, non-compressed, mixture of the P1 ingredients was also obtained for characterisation purposes. For P2, a 5 mm thick sheet of the prepared pavement material was broken up into crumb sizes of about 10 mm diameter to undertake characterisation.

A composite sample of each of P1 and P2 was separately prepared with three random subsamples collected from the bulk crumbed mixtures with a stainless-steel trowel. The composite samples were sent to a NATA-accredited commercial laboratory to characterise the products for relevant EOL tyre rubber CoPC (Table S1). NATA is the National Association of Testing Authorities, which is the recognised national accreditation authority for analytical laboratories and testing service providers in Australia.

2.2 Leaching experiments

A standardised agitated batch laboratory leaching test (ASLP) and the new SSLP were conducted to understand the leachability of the CoPC from the two products (P1 and P2).

2.2.1 ASLP. Developed to somewhat mimic leachability in landfills, the ASLP[14] was followed as a standard batch test for comparison to the new SSLP leaching method. ASLP batch testing consisted of a 20 : 1 ratio of crumbed pavement mixture to water (100 g of solid and 2 L of neutral pH ultrapure water (Milli-Q®)) in a 4 L glass jar agitated on an end-over-end shaker at 30 rpm for 20 h at ambient temperature (25 °C). After agitation for 20 h, the supernatant was separated from the solid without centrifugation and filtration, stored in another glass jar and subsequently analysed for CoPC at the same NATA-accredited laboratory where the total chemical analysis and the SSLP samples were conducted. The leached chemical concentrations were also used to calculate the percentage mass removed during leaching compared to the initial mass in the samples.

2.2.2 SSLP – new static surface (core) leaching procedure. The new SSLP method was designed to mimic in the laboratory (within practical limits) the likely leaching of chemicals from pavements and flat surfaces. We did not seek to precisely mimic variations in rainfall volume or intensity, as the intent was to evaluate a method that might be more routinely applied in a commercial laboratory setting – much like ASLP/LEAF methods.

The leaching was conducted using the whole core sample as shown in Fig. 1. The SSLP design was established to ensure largely one-dimensional (1-D) leaching from the surface of the core, as would be the case for a laid-down pavement of P1 or P2 during rain events. The design implemented consisted of a wide-mouthed 4 L glass jar (17 cm diameter and 24 cm high) with 750 mL ultrapure water (Milli-Q®). The cores (10 cm diameter and 5 cm high) were hinged tightly in a stainless-steel clamp, hung using stainless-steel wire, and suspended in ultrapure water. The core was submerged by approximately 5 mm (Fig. 1). Neither the clamp nor the wire was in contact

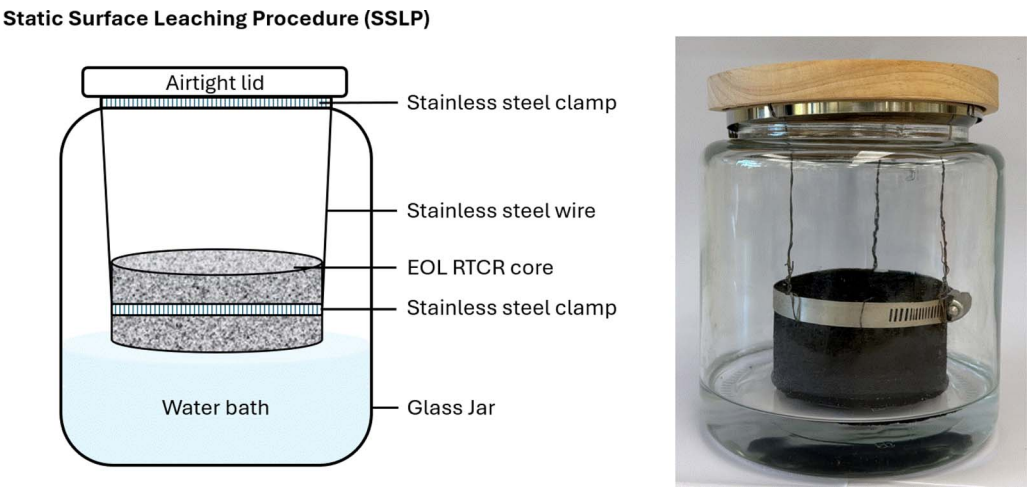

Fig. 1 Schematic and image of the experimental set-up for the static surface (core) leaching procedure (SSLP).

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with the water. Each static core leaching experiment was conducted in triplicate.

As planned, no advective transport or mixing of water occurred during the SSLP tests. As such leaching was induced largely due to one-dimensional diffusional transport of CoPC from the core surface into the water. This is later shown to be validated by the data. The characteristic time for diffusion to operate and stabilise over the entire core thickness that was immersed can be approximated as L^2/D, where L is the thickness of the core below the water (5 mm), and D is the diffusion coefficient. For some organic solutes the range of diffusion coefficients in water are 0.6×10^{-5} cm^2 s^{-1} for benzo(*a*)pyrene and 1.8×10^{-5} cm^2 s^{-1} for methanol.[38] In a water saturated porous media, depending on the porosity, this might reduce by an order of magnitude or more. As such the characteristic time for chemical release rates to deplete or stabilise from 5 mm of the core becomes approximately 1–12 months. It indicates that the 5 mm section of core would release chemicals slowly but would not be depleted over the time of the experiments, when extended out to 14 d.

Though the SSLP core experiments were designed to avoid full immersion and the need to seal surfaces, as required by LEAF 1315, to align SSLP with a standard leaching approach, the principles and time stepping advised in LEAF 1315 were followed.[10] LEAF 1315 describes the leaching of inorganic CoPC from a monolithic material into water whereby the surface or body of the monolithic material is exposed to water, usually *via* immersion of the entire monolith or one of the monolith surfaces in a water bath with or without sealing of surfaces of the monolith. Aligned with LEAF 1315, the volume of water used (750 mL) was calculated based on the area of the flat surface of the core that was in contact with water using the approximation 9 ± 1 mL per cm^2, similar to LEAF 1315. The water remained static in the jars (unagitated) for the duration of the leaching experiment. The glass jar was covered with aluminium foil to prevent exposure to light throughout the experiment and was at ambient temperature. A new (clean) ultrapure water (Milli-Q®) volume was replaced to start the next period of leaching at LEAF 1315-aligned intervals of 2 h, 24 h, 48 h, 7 d and 14 d, respectively, and samples for leachate analysis were collected at these time points prior to water replacement.

Each water sample was decanted into another glass container after the duration of leaching ensuring a well-mixed water volume prior to subsampling for analysis. During storage prior to analysis, the glass container was also covered with aluminium foil to prevent exposure to light throughout the experiment and kept at 4 °C. A subsample of the water volume was used to measure leachate pH and EC, and another subsample was sent to the NATA-accredited laboratory for analysis of CoPC in the leachate. The collected samples were dispatched to the NATA-accredited laboratory in two separate batches – the first included samples obtained at 2 h, 24 h, and 48 h, while the second comprised those collected at 7 d and 14 d. The leached concentrations were also summed across the five leaching events and used to calculate the total percentage mass removed during leaching compared to the initial mass in the samples.

2.3 Analytes

Based on prior literature and known likely chemicals in RTCR, a range of CoPC were analysed, across 80 analytes. These included metals, hydrocarbons, halogenated and non-halogenated phenols, phthalates, triazoles, and other tyre additives deemed to be of concern, such as 6PPD-Q, HMMM and 1,3-DPG (Table S1). The limit of reporting (LOR) is the lowest amount of an analyte in a sample that can be quantifiably determined with acceptable precision and accuracy under the stated analytical conditions (also called the lower limit of quantification or LOQ).

3 Results and discussion

3.1 Total chemical analysis concentrations

The complete dataset for the total and batch-leachable concentrations for the P1 and P2 materials are presented in Tables S2 and S3, respectively. The P1 material had higher calcium and magnesium concentrations (74 000 and 14 000 mg kg^{-1} compared to 1500 and 3900 mg kg^{-1} respectively in P2), comparable total recoverable hydrocarbons (TRH) concentrations, and lower PAH concentrations (<2 compared to 5 mg kg^{-1}) to those in P2. P1 had a broader range of metals than P2, including arsenic, lead, lithium, and nickel found in P1 above the LOR. This may be due to the broader range of materials used in manufacturing P1 (including sand and bitumen) compared to P2. The highest metal concentrations were for aluminium and iron.

Of the tyre additives and their transformation products, 1,3-DPG and HMMM were not detected above LOR in the P1 samples, but 6PPD-Q was found at 96 μg kg^{-1}. In contrast, 1,3-DPG was 100 μg kg^{-1}, 6PPD-Q was 3000 μg kg^{-1} and HMMM was 140 000 μg kg^{-1} in P2. This likely reflects the 10-fold higher ratio of RTCR in P2 compared to P1.

3.2 ASLP batch leaching results for P1 and P2

Tables 1 and 2 summarise all data for P1 and P2, respectively, where the analyte was above LOR (mg kg^{-1} or μg kg^{-1}) in the total analysis, and at the same time found to be above LOR (mg L^{-1} or μg L^{-1}) in the ASLP batch-leachable or SSLP fractions. Of nearly 80 analytes (see Tables S2 and S3), for P1, only five analytes had total and ASLP leachable concentrations above LOR, and for P2 there were only four analytes.

The percentage mass leached from P1 in the ASLP batch tests relative to the total concentration ranged from a low of 0.05% for TRH up to 1.27% for 6PPD-Q. These indicate that a substantial mass of each chemical remained in the sample under this reasonably aggressive agitated ASLP test.

Whereas, P2 had mass fractions of some analytes that leached up to 34% of the total mass (Table 2). The three rubber anti-degradant/antioxidant compounds were detected in and leached from P2. For 1,3-DPG, approximately 34% of the measured total mass was leachable at a concentration of 1.7 μg L^{-1}, and approximately 30% of HMMM was leachable at a concentration of 2100 μg L^{-1}. Only 0.33% of 6PPD-Q was leachable at a concentration of 0.49 μg L^{-1}, which is lower than that found for P1. None of the TRH fractions were found to be

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Table 1 Total, ASLP batch leachable and maximum of the mean concentrations determined during the SSLP core test for P1. Data are only shown where the analyte concentration was above LOR in the total and either of the ASLP or SSLP leachate analysis (complete data are in the SI). Percent of the mass leached during the ASLP and SSLP tests are also shown

	Total concentration (mg kg^{-1})	ASLP leached concentration (mg L^{-1})	% leached (%)	SSLP – maximum of means (mg L^{-1})	% leached (%)
Calcium	74 000	3.9	0.11	5.6	0.01[a] (0.1[b])
Aluminium	4600	0.41	0.18	<0.05	
Copper	6.7	<0.01		0.005	0.09[a] (0.9[b])
Iron	1100	0.17	0.31	<0.001	
Zinc	73	<0.01		0.031	0.02[a] (0.2[b])
TRH > C10–C40 (total)	4400	0.12	0.05	<0.1	
	µg kg^{-1}	µg L^{-1}	%	µg L^{-1}	%
6PPD-quinone	96	0.061	1.27	0.025	0.05[a] (0.5[b])

[a] Based on the total concentration/mass of the whole core. [b] Based on concentration/mass of 10% of the core.

leachable from P2. Leachate concentrations with respect to guidance criteria are discussed further.

3.3 SSLP (new method) leachable concentrations

The mean and standard deviation of concentrations of three replicates for each of the five leaching periods are presented in Table S4 for P1 and Table S5 for P2. The maximum of the mean concentration for the triplicates across all the five leaching periods are included in Tables 1 and 2, for cases where leachate concentrations and total chemical concentration were both above LOR.

Fig. 2 shows 6PPD-Q concentration as an example of solute concentration changes observed after each increase in the leaching period. Data for each of the individual triplicate cores for P1 and P2 show minimal variation across the replicates. At the scale represented, no variation is observable on the P1 core data. As might be expected, concentrations largely increased as the leaching period extended from 2 h to 14 d. Notably, the leachate concentrations of 6PPD-Q from ASLP were higher than those from the SSLP at the equivalent period (of approximately 1 d). For P2, the ASLP concentrations were lower than those observed for SSLP at 7 and 14 d.

For P1, the mean concentrations of leached calcium increased monotonically from <0.5 mg L^{-1} for the 2 h leaching duration up to 5.63 ± 0.46 mg L^{-1} after the longest leaching duration of 14 d (Table S4). In contrast, the mean sodium concentrations did not vary much regardless of the leaching duration. Similarly to the ASLP test, magnesium did not show a leachable concentration above the LOR throughout the study despite having a relatively high total concentration. In contrast to the ASLP test, which showed only aluminium and iron concentrations above LOR (Table 1), for the static pavement method (Tables S4 and 1) copper and zinc were the only metals that had concentrations above LOR, albeit variable across the leaching durations. There was no leaching of detectable concentrations above LOR for bisphenol A (BPA), phenols (halogenated and non-halogenated), phthalate esters, PAHs and triazoles (Table S4). TRH fractions were largely not leachable, with only one detection above LOR at 2 h. The mean

Table 2 Total, ASLP batch leachable and maximum of the mean concentrations determined during the SSLP core test for P2 (containing an order of magnitude greater proportion of RTCR). Data are only shown where the analyte concentration was above LOR in the total and either of the ASLP or the SSLP leachate analysis (complete data are in the SI). The percentage of the mass leached during the ASLP and SSLP tests are also shown

	Total concentration (mg kg^{-1})	ASLP leached concentration (mg L^{-1})	ASLP – % leached (%)	SSLP – maximum of means (mg L^{-1})	SSLP – % leached (%)
Copper	22	<0.01		0.007	0.16[a] (1.6[b])
Zinc	1500	0.23	0.31	0.020	0.003[a] (0.03[b])
TRH > C10–C16	130	<0.05		0.367	1.21[a] (12.1[b])
TRH > C10–C40 (total)	3570	<0.1		0.400	0.05[a] (0.5[b])
TRH > C16–C34	2800	<0.1		0.100	0.004[a] (0.04[b])
	µg kg^{-1}	µg L^{-1}	%	µg L^{-1}	%
1,3-DPG (1.3-diphenyl guanidine)	100	1.7	34	0.72	2.88[a] (28.8[b])
6PPD-quinone	3000	0.49	0.33	0.82	0.12[a] (1.2[b])
HMMM (hexamethoxymethylmelamine)	140 000	2100	30	243	0.62[a] (6.2[b])

[a] Based on the total concentration/mass of the whole core. [b] Based on concentration/mass of 10% of the core.

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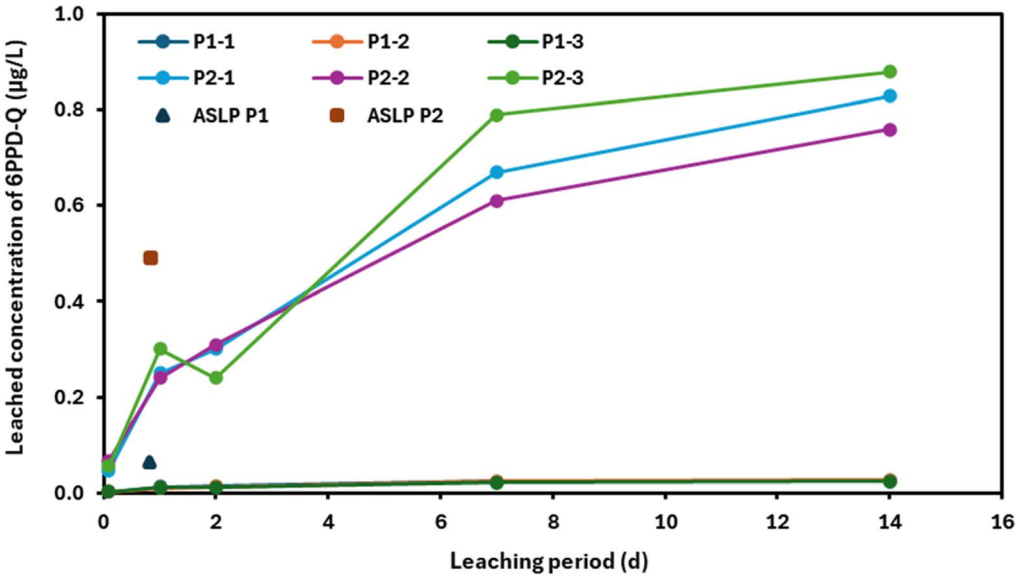

Fig. 2 Time series of 6PPD-Q concentrations measured at the end of each leaching period using the SSLP approach (data points connected by lines) and corresponding ASLP concentrations at the equivalent leaching duration (~1 d, shown as single data points). SSLP results are presented for each of the triplicate cores of P1 and P2, denoted P1-1 to P1-3 and P2-1 to P2-3, respectively.

leachable concentration of 6PPD-Q increased gradually from 0.0018 µg L^{-1} at 2 h up to 0.0250 ± 0.0010 µg L^{-1} for the maximum 14 d period. 1,3-DPG and HMMM were only found above LOR at the 48 h leaching period.

For P2 (Table S5), in contrast to P1, calcium was not above LOR for any of the five leaching periods, and sodium appeared depleted after two leaching periods reducing to <0.5 mg L^{-1} at 48 h and longer leaching durations. Similarly to the P1 results, copper and zinc were leached from P2 at above LOR concentrations at most leaching events, and there was no leaching of detectable concentrations above LOR for BPA, phenols (halogenated and non-halogenated), phthalate esters, PAHs or triazoles (Table S5). Leached concentrations above LOR were measured for most TRH fractions (Table 2). The TRH was largely dominated by carbon ranges >C10–16, which increased with an increase in the leaching duration from <0.01 mg L^{-1} to 0.37 mg L^{-1} (Table S5). Likewise, the concentration of both 1,3-DPG and 6PPD-Q increased with increasing leaching duration from 0.07 µg L^{-1} at 2 h to 0.70 µg L^{-1} at 14 d for 1,3-DPG and 0.06 µg L^{-1} at 2 h to 0.80 µg L^{-1} at 14 d for 6PPD-Q. For 6PPD-Q the minimum–maximum concentration range leached from P2 was about 30 times those from P1. The mean leachable concentration of HMMM ranged up to 243 µg L^{-1} at the 14 d leaching duration, but with a large standard deviation across triplicates. Given the leached concentrations at 2 h, 24 h and 48 h ranged from 54 to 130 µg L^{-1} and are the same order of magnitude as that at 14 d, the 0.5 µg L^{-1} value at 7 d appears anomalous. It is known that HMMM can break down,[39,40] so if holding times are protracted, mass can be lost. In this case, the sample collected at 7 d had a holding time of approximately 14 d and this may have compromised the HMMM data at this time point. All other P2 samples were analysed within 3–5 d of collection.

Also included in Tables 1 and 2 (expanded in Tables S4 and S5) is the cumulative mass removed over the five SSLP leaching durations as a percentage of the total initial mass. We note that the total monolith was not immersed and as such only a tenth of the total mass was likely available to be leached (only 5 mm of a 5 cm length of core). Percentages based on both estimates of available mass are in Tables 1 and 2. Most percentage masses removed across the analyte range were extremely low. The highest for SSLP using the total mass available in the core was 2.88% for 1,3-DPG (for P2 in Table S5), in contrast to the 34% removal of 1,3-DPG mass during the ASLP test with active agitation over 20 h (Table 2). If we assume only 10% of that core mass was available however, then the percentage leached mass increases to ~29% which then becomes comparable to the ASLP test. The percentage estimates overall, affirm that mass depletion from the cores evaluated in the SSLP leaching method was not likely to play a role in limiting mass diffusion from the cores into the water bath for each leaching period.

To further elucidate the underlying processes under SSLP conditions, example data for the mass flux and cumulative mass leached for 6PPD-Q are presented in Fig. 3. Data for all triplicate P1 and P2 cores are shown and normalised to the common surface area of the core and plotted on a log–log scale. The mass flux (Fig. 3a) exhibits a decreasing trend over time, consistent with the gradual increase in solute concentration in the water bath, which reduces the concentration gradient between the core and the leachant. The cumulative mass of 6PPD-Q leached over the time normalised to the surface area (Fig. 3b), on a log–log plot effectively approximates a straight line with an estimates slope of near 2 : 1. This is the ratio that might be expected (based on Crank[41]) if one-dimensional diffusion from the core into the water bath was the primary process governing the

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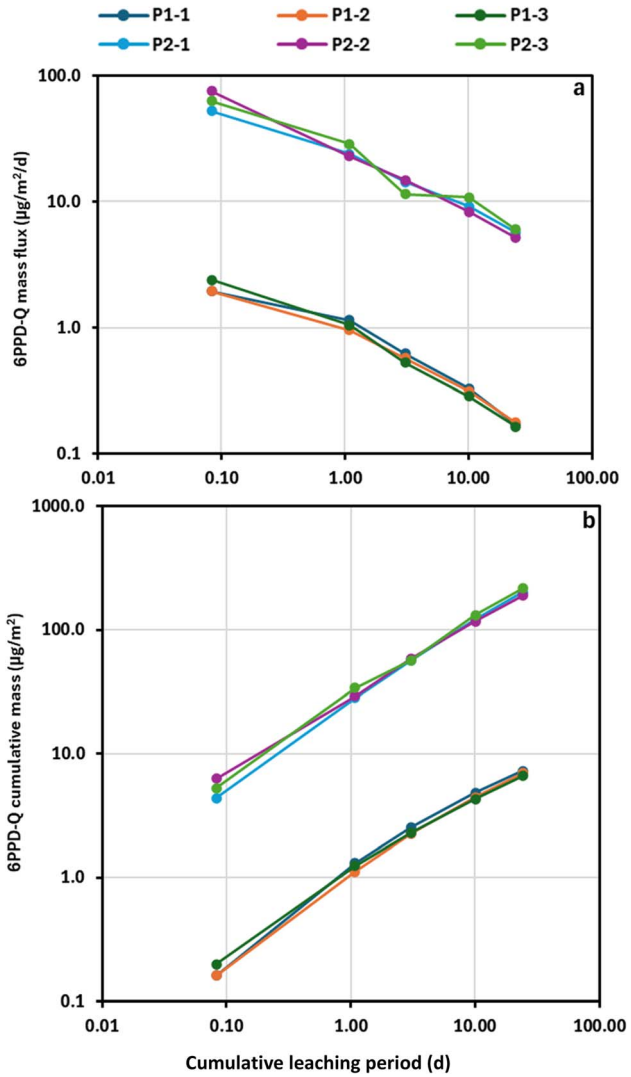

Fig. 3 Mass flux (a) and cumulative mass (b) of 6PPD-Q over the cumulative leaching period obtained from the SSLP approach. Data are presented for each of the triplicate cores of P1 and P2, denoted P1-1 to P1-3 and P2-1 to P2-3, respectively.

leaching, as pointed out by Kosson *et al.*[26] when describing LEAF 1315 data.

3.4 Synthesis and comparison to concentration criteria

Whilst the ASLP test involves agitation to maximise leached concentrations for risk assessment and potential disposal of hazardous materials, not all ASLP leached concentrations were higher than those found from the SSLP method. This may in part be due to extended leaching times in the SSLP tests beyond the 20 h period used in the ASLP test (as in Fig. 2 for 6PPD-Q). For example, the copper concentration was <LOR in the ASLP leachate for both P1 and P2, whereas the SSLP leachate had a maximum mean concentration above LOR, albeit still at low concentrations. Also, the zinc concentration was highest in SSLP leachate from P1 (0.031 mg L^{-1}) as compared to the ASLP leachate which was below the LOR. In contrast, the zinc concentrations were lower for SSLP for P2, as compared to the ASLP leach. For P2, a broad range of TRH fractions were detected above LOR during the SSLP tests, compared to below LOR concentrations for ASLP. Across the tyre additives and associated chemicals, if detected in leachate from the ASLP, they were also observed in leachate from the SSLP. It could be argued, however, that if we are suggesting use of the SSLP method to better represent the likely leachable compounds and their concentrations in water in contact with field-installed pavements, then less extensive durations of water contact might be best adopted – such as 2 h or 1 d, rather than 14 d. Contrary to this might be applications where water is ponded on pavement surfaces, such as boat ramps or other low-lying environments combined with high water tables.

Where available, a brief comparison of the concentration of CoPC to guidance and threshold concentration criteria is given – with some values tabulated in Tables S3 and S4. It should, however, be noted that in the environment, leached concentrations will further dilute. The ASLP leachable concentrations of metals detected in P1, except for aluminium (thresholds of 0.8–27 µg L^{-1}), were below the respective Australian threshold values.[42] For P2 (Table S3), the ASLP leachable concentrations of all metals, except for zinc (threshold of 2.4 µg L^{-1}), were deemed safe for the protection of 99% freshwater species.[42] In contrast, the SSLP leachable concentrations for copper and zinc from both P1 (Table S2) and P2 (Table S3) were above the ANZECC/ARMCANZ threshold values for the protection of 99% freshwater species for copper (1 µg L^{-1}) and zinc (2.4 µg L^{-1}).[42] Note, the ANZECC/ARMCANZ threshold concentrations for the protection of aquatic ecosystems are default guideline values or trigger values for the protection of ecosystems in Australia. Measured concentrations above the trigger value in leachate should trigger a management response, but do not necessarily mean that the receiving aquatic ecosystem will be adversely affected.

Tables S6 and S7 contain references to published toxicity study concentrations for the tyre additive 1,3-DPG and 6PPD-Q, respectively. This comparison is intended to provide a preliminary risk characterisation under controlled laboratory conditions, using USEPA[43] ECOSAR-predicted LC50 (lethal concentration, 50%) values and relevant literature benchmarks. Tables S8 and S9 present LC50 values for 1,3-DPG and 6PPD-Q predicted by ECOSAR.[43] Traces of 1,3-DPG (close to the LOR of 0.05 µg L^{-1}) were detected when the sample P1 core was in contact with water for 2 h and 48 h intervals but otherwise was not detectable above LOR. Previous studies have shown that a concentration of 1,3-DPG ranging from 0.75 to 30.6 mg L^{-1} can cause toxicity to *Daphnia magna* and a concentration ranging from 2.88 to 28.5 mg L^{-1} can be toxic to *Tetrahymena pyriformis*[44] (Table S6). However, the LC50 values (neutral organics) predicted by ECOSAR[43] are 27.31 mg L^{-1} for fish and 16.84 mg L^{-1} for daphnids (Table S8). For P2, the ASLP leachate concentration was 1.7 µg L^{-1}, several orders of magnitude lower than the ECOSAR-predicted LC50 values (Table S8) and concentrations of concern reported in previous studies.

6PPD-Q leached from both P1 and P2 with or without aggressive agitation, *i.e.*, for both ASLP and SSLP methods. For

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both P1 and P2, the 6PPD-Q concentration increased with the SSLP leaching duration. These results demonstrated the potential for greater leaching of 6PPD-Q over time and under less aggressive conditions, although it could be argued that rarely would ponded-water persist on paved areas for extended periods like 14 d to allow such enhanced leaching. In addition, the maximum mean concentration of 6PPD-Q leached during the SSLP tests of P2 material was close to twice the concentration of the ASLP leachate concentration, but again shorter duration leachates from the SSLP test had lower leachate concentrations than ASLP. Toxicity studies for 6PPD-Q exposure across various aquatic species revealed significant differences in the toxicity values of several orders of magnitude (0.041–2200 $\mu g\ L^{-1}$)[28,29,31–33,35–37,45] (Table S7), indicating varying sensitivity to environmental conditions and exposure durations. For P1, the maximum SSLP leached concentration of 6PPD-Q (0.025 $\mu g\ L^{-1}$) was only higher than one of the sixteen toxicology study values noted in Table S7, whereas for P2 the maximum SSLP leached concentration (0.82 $\mu g\ L^{-1}$) was higher than four of the values. This varying toxicity within the literature is important, indicating the need for additional toxicity studies with country-relevant species. Notwithstanding the concentrations reported in the literature, the ECOSAR-predicted LC50 values for 6PPD-Q are 40 $\mu g\ L^{-1}$ for fish and 90 $\mu g\ L^{-1}$ for daphnid (Table S9), which are 2–3 orders of magnitude above the concentrations observed in this study.

HMMM was either not detected (LOR = 0.05 $\mu g\ L^{-1}$) or, if detected, was at a very low concentration in P1 SSLP leaching test results. The P2 sample showed ASLP leached concentrations of 2100 $\mu g\ L^{-1}$, and mean SSLP leached concentrations up to 243 $\mu g\ L^{-1}$. This is lower than the LC50 (lethal concentration, 50%) (neutral organics) predicted by ECOSAR[43] of 725 mg L^{-1} for fish and 397 mg L^{-1} for daphnids (Table S10). Little Australian data exists on the toxicity of HMMM on relevant Australian species. Whilst Rauert *et al.*[46] investigated HMMM in Australian waterways, no toxicity effects studies were undertaken.

We recognise that the toxicity assessments cited here are chemical specific but that the leachates generated from the RTRC reuse products are complex mixtures, consisting of metals, TRH, and other additives. Apart from the need for country-specific ecological effects studies, evaluating toxicity from likely or otherwise relevant leachable mixtures seems warranted. This might be more important when the leached concentrations for specific analytes are less than threshold or trigger values, whereby the additive effects of mixtures may hold greater risks.

3.5 Limitations and future research

The SSLP core leaching method was developed to have water come in contact only with the surface of a cored sample (rather than a monolith being immersed entirely in water as in the LEAF 1315 approach). As such, SSLP better reflects the potential leaching from the surface at a field installation of the rubberised pavement materials. We used the same pavement cores for water sampling across different time points. Our rationale was to enable SSLP outcomes to be directly aligned with and comparable to different time point samples from LEAF 1315,[10] and to track mass losses over time. Moreover, our approach also replicates real-world conditions where surface areas are repeatedly exposed to water, allowing for cumulative leaching. We note that although destructive sampling using fresh materials for each sampling event could help isolate leachate from untouched surfaces, our approach aimed to assess the leaching behaviour under repeated exposure, which may be more representative of field scenarios. We also note that the fraction of chemicals leached during earlier time points was limited compared to the total content present in the material, and leaching predominantly occurred at the surface and according to diffusional transport from the pavement materials into the water bath. We recommend that future studies compare results from both approaches to better understand the impact of sampling strategy on leachate composition.

A limitation of the current approach is uncertainty on the timeframes over which leaching should be pursued. Another potential limitation could be the wicking effect/capillary action that may result in water being absorbed into the core, effecting the experimental water volume and leaching ratios. Submerging the core by about 5 mm might lead to depletion of CoPC in that zone creating mass transfer limitations to the water phase, although this might be less likely for shorter intervals during the leaching test. A modification to the leaching set up could be to seal the outer surface around the circumference of the core and the bottom surface and have the water bath sit on the top surface of the core much like that proposed in the LEAF 1315 approach. Complications with this alternate approach might be (i) choosing a sealant that does not react with or add to any of the CoPC present in reuse materials or other pavement contaminants, and (ii) ensuring the core is sealed adequately within the test container so water does not bypass the top surface of the sealed core sample. In seeking a routine leaching protocol, usable in commercial laboratories, much like ASLP and the LEAF protocols, the proposed SSLP static protocol for pavements addresses these limitations by simplistically avoiding sealing issues entirely.

While the SSLP assessment method used here was developed to approximate the *in situ* field use of the pavement material, no direct comparison in the field under natural rainfall conditions or run-off has yet been undertaken. A direct comparison of the two would provide a more compelling justification for the use of this modified approach and provide greater confidence that the leached fractions are representative of field conditions. Additionally, laboratory testing of 'aged' pavement materials or measurements in the field over time would give insights as to the effect of weathering in reducing the release of leachable fractions and concentrations. Similarly, altering the temperature of the water bath would enable greater confidence in the findings across different climate zones.

The in-field application and use of the rubberised pavement materials might also alter which leaching method might be more appropriate. The ASLP method, while more aggressive in agitating the sample, might be appropriate where the pavement is exposed to high-impact activities such as when used in high-

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volume traffic road resurfacing, or possibly where the pavement is constantly or frequently inundated (*e.g.*, high groundwater tables, flood plains, or near wetlands).

We note too that tyres can be sourced globally and, thus, EOL tyre material is highly heterogeneous. Whilst testing was run in triplicate, samples containing differing source tyres would likely yield altered results. Nevertheless, these results are consistent with results from parallel testing of EOL tyres prior to their remanufacture for reuse, providing confidence that the results can be relied upon as an indicator of the types of chemicals found in and potentially released from tyre materials upon exposure to water in the environment. Different binding agents are also used in the differing reuse pavement materials – we did not seek to undertake an analysis of binding agents, or the amounts used in the P1/P2 materials.

3.6 Relevance to PFAS leaching from asphalt and concrete

As indicated, PFAS in asphalt and concrete is an active research focus.[16–19,47–50] When assessing the leaching of PFAS from intact concrete and asphalt pads and pavements, laboratory methods largely rely on LEAF and ASLP procedures, although alternates have been suggested for leaching of PFAS from soils to better mimic natural field conditions.[22,48] All laboratory methods largely involve immersion in water of monoliths or having materials broken down or ground to set particle sizes. The currently proposed static leaching method (SSLP) involving contact of water with the surface of the intact cored material could be further evaluated to mimic asphalt/concrete paved surfaces and what concentrations of PFAS might be washing off such surfaces over time when left *in situ*.

4 Conclusions and implications

A new method for measuring the leachability of CoPC from pavement materials under passive, and more realistic exposure conditions, was developed and compared against standard methods. As compared to the existing methods such as ASLP and LEAF, SSLP enables water contact only with the material surface, offering a realistic simulation of on-site exposure scenarios while remaining practical for use in commercial laboratories and provides a foundation to appropriately assess the availability of identified CoPC from pavement materials that contain recycled content. It was shown that leaching using the SSLP approach approximated one-dimensional leaching from the pavement surface, and was dominated by diffusive processes, as was desired to yield a simple repeatable approach.

Two pavement materials manufactured with markedly different (by an order of magnitude) recycled tyre rubber crumb content were evaluated using ASLP and SSLP. Generally, the SSLP indicated lower leached concentrations than the ASLP, especially for shorter duration (<2 d) SSLP, reflecting the reduced agitation and surface-specific contact typical of field conditions. The product with the highest proportion of RTRC led to the leaching of some chemicals at concentrations that may have adverse effects on some biota, reinforcing the need to identify acceptable RTRC proportions for reuse applications and to further investigate binder influences on leaching outcomes.

Although this study replicated cumulative exposure through repeated leaching of the same samples, future work should examine field conditions directly, particularly the influence of rainfall, weathering, temperature, and ageing on leachate composition, and evaluate alternate sampling strategies that isolate pristine surfaces. Comparative assessments between SSLP, LEAF, and ASLP approaches would clarify protocol sensitivity and strengthen confidence in laboratory-to-field extrapolations.

Finally, as SSLP focuses on intact cores, it could be extended to assess the leaching of PFAS and other persistent compounds from asphalt and concrete surfaces. The method provides a practical foundation for regulators and industry to advance circular economy goals by supporting reliable environmental assessments of products containing recycled materials.

Author contributions

Prashant Srivastava: conceptualisation, methodology, investigation, writing – original draft, writing – review & editing, project administration, funding acquisition; Mitzi Bolton: writing – review & editing, conceptualisation, funding acquisition; Naomi J. Boxall: writing – review & editing, conceptualisation, funding acquisition; Sonia Mayakaduwage: investigation; Avanthi Igalavithana: investigation; John L. Rayner: methodology; Greg Davis: writing – original draft, writing – review & editing, conceptualisation, methodology, funding acquisition.

Conflicts of interest

Greg Davis reports financial support was provided by Australian Government Department of Climate Change, Energy, the Environment and Water – Supervising Scientist Branch. Other authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

The data supporting this article have been included as part of the supplementary information (SI). Supplementary information is available. See DOI: **https://doi.org/10.1039/d5em00516g**.

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