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Simultaneous sampling for microplastics and environmental contaminants from artificial turf: development of a new integrated microplastics eluate lysimeter

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Abstract

To get a better understanding of potentially harmful contaminant emissions from soils or materials into the environment, politics demand practical and holistic sampling concepts for environmental samples such as leachates containing polycyclic aromatic hydrocarbons (PAH) or heavy metals, and hazardous particulate matter like microplastics (MP). Of particular concern are MP emissions from artificial turf sports pitches. So far, there has been only very limited data on MP mass emissions from artificial turf potentially posing a risk to the groundwater and no sampling device that allowed simultaneous sampling for dissolved and particulate contaminants. In this study, a novel integrative microplastics eluate lysimeter was developed to determine contaminant emissions from three artificial turf systems at different ageing states (fabric-new, artificially aged, real-time aged). For the accelerated ageing, all environmental simulation parameters were based on Central German conditions and simulated outdoor stress during the turf service lifespan of 15 years. MP masses from eluates were analysed using thermal extraction desorption-gas chromatography/mass spectrometry, PAH concentrations using gas chromatography/mass spectrometry and heavy metals using inductively coupled plasma-optical emission spectroscopy. Results showed that no PAH or heavy metal concentrations from the seepage water were above German legal limits for alternative granular construction materials considering soil and groundwater protection. Furthermore, it was found that only minimal MP emissions were released from new turf systems into the seepage water (< 1 mg/m^2). Ageing of the artificial turf increased MP formation, especially from rubber infill and grass fibres, which are then carried into the seepage water. The highest total MP emissions over a simulated turf lifespan of 15 years were detected in two real-time aged turf systems ranging from 136.4–252.5 mg/m^2. Considerably less total MP emissions were detected in accelerated aged artificial turf systems, one of which contained a synthetic rubber infill (5.4–8.0 mg/m^2) and one without rubber infill (0.2–5.3 mg/m^2). In summary, it was demonstrated that the newly developed MEL generated reliable and reproducible data and has thus proven itself as an integrated, straightforward and automated sampling device for simultaneous monitoring of particulate and dissolved pollutant emissions from simple soil matrices.

Keywords Lysimeter, Microplastics, Artificial turf, Thermal extraction desorption-gas chromatography/mass spectrometry, TED-GC/MS, Polycyclic aromatic hydrocarbons, PAH, Heavy metals, Soil samples

Background

Public and political awareness of potentially harmful emissions of man-made plastics into the environment is steadily increasing. In addition to health concerns about released pollutants (e.g. polycyclic

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aromatic hydrocarbons, PAH), particulate matter such as microplastics (MP) is also constantly gaining in relevance [12, 46, 54]. Based on the precautionary principle, the European Commission has therefore introduced the new Directive 2023/2055 in September 2023 to reduce MP emissions into the environment, which includes the sale and use of intentionally added MP [33]. This ban explicitly applies to the use of synthetic rubber infill in artificial turf systems because of its particle size < 5 mm. However, the ban also raises concerns about the impact on football clubs and recreational sport and thus the future of artificial turf sports pitches.

In general, the amount of artificial turf pitches in Europe is steadily increasing. This is because artificial turf has established itself in recreational sports due to many advantages. Compared to natural turf, artificial turf requires less maintenance (e.g. fertilisation, watering) and is playable throughout the year in almost any weather condition. Therefore, it allows for intensive usage, which is particularly crucial to cover the demand for recreational sports in high-density living areas. According to the ECHA, there were 13,000 artificial turf pitches in the EU in 2019 [31]. There is no official data on the number and area of artificial turf pitches in Germany [14]. However, it was estimated from the DIN NA 005–01–22 AA Working Committee on Artificial Surfaces and Artificial Turf Surfaces in 2019 [24] that there are a total of around 7,000 artificial turf pitches in Germany, varying in surface area and infill type. Most of them (ca. 5,000 pitches) are large pitches with an average size (ØA) of 7,000 m^2, of which ca. 3,500 pitches are filled with rubber granules, ca. 1,000 pitches are purely sand filled and ca. 500 are unfilled hockey pitches. In addition, there are approx. 200 small pitches (ØA: 700 m^2) and 1,400 mini pitches (ØA: 260 m^2), which are also filled with rubber granulates. Presently in Germany, synthetic infill made of EPDM (ethylene propylene diene monomer rubber, Fig. 1c) is most commonly used. EPDM is considered to be more environmentally friendly than the formerly used granules made from shredded end-of-life car tyres, which include, among others, SBR (styrene butadiene rubber) and can partially contain hazardous additives, e.g., heavy metals like zinc and lead or PAH [2, 31, 44]. Synthetic rubber infill, or the so-called performance infill, is added to artificial turf to protect players from injuries and the grass fibres and backing from abrasion. It improves material longevity to maximise the artificial turf service lifespan, which is typically around 12–15 years. In general, artificial turfs for sports pitches are highly developed and complex multi-component systems and consist of multiple synthetic polymers from which MP can potentially emit. Especially, abrasion of grass fibres is also considered to be a relevant MP source [13]. The calculation of an overall mass balance for MP emissions from artificial turf pitches is very complex, as the individual site-specific conditions can vary greatly from pitch to pitch. The following parameters are important: i) climate zone (Nordic countries show a much higher discharge due to snow); ii) generation of artificial turf (third-generation systems show reduced discharge behaviour); iii)

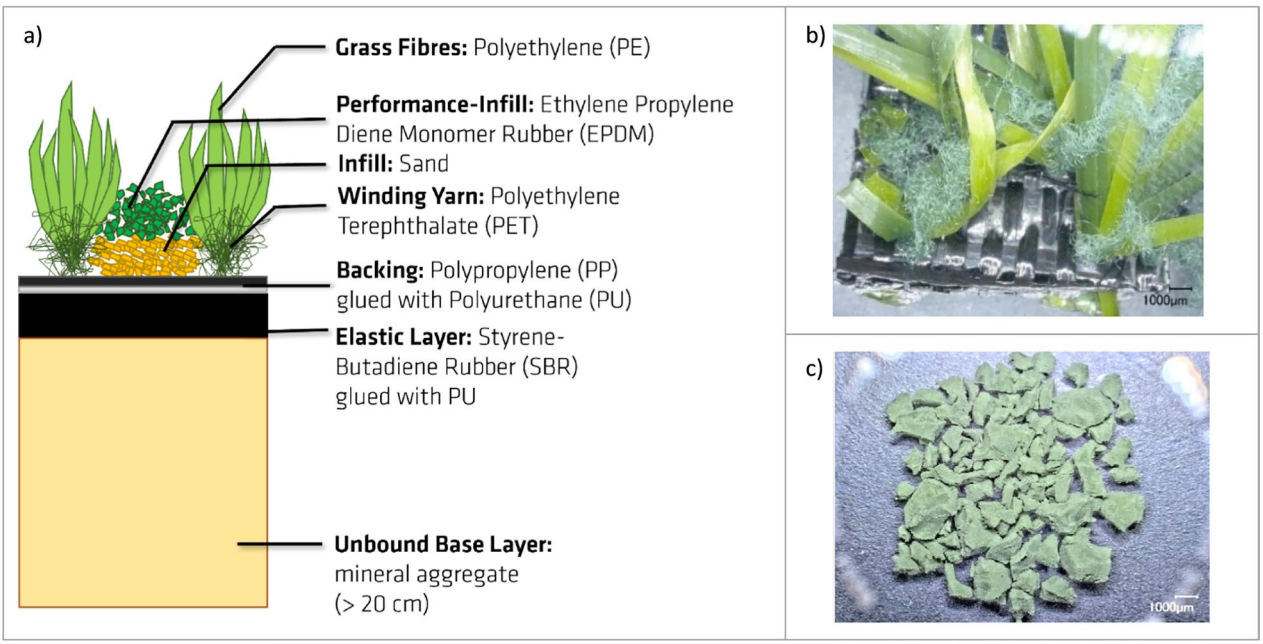

Fig. 1 Artificial turf composition: **a**) schematic overview, **b**) microscopic picture of artificial turf and **c**) of EPDM granules

structural installations (with/without drainage system, type and quantity of infill). There have already been various studies from a wide variety of organisations in the past, mainly focusing on the emissions of rubber granules [4, 47, 48]. Based on surveys of associations, local authorities and experts in planning, construction, supply and production, as well as the experience of its members, the DIN NA 005–01–22 AA (2019) estimated that approx. 60–80% of granule losses are due to a lack of or improper maintenance measures and techniques, approx. 5–15% to snow clearing, approx. 10–20% to heavy rainfall events and wind drift and approx. 3–5% to adhesion to athletes' clothing, bodies and shoes. Depending on the construction of the artificial turf pitch, the seepage water is either passed through a drainage system into a sewage treatment plant or surface water, or it infiltrates through the soil into the groundwater.

So far, to our best knowledge, there is no reliable data to estimate total MP emissions from seepage water from artificial turf sports pitches. Furthermore, no holistic sampling concepts and devices are available to allow simple and fast analysis of dissolved and particulate contaminants in eluates from soil media [39]. However, this is of great political interest and regulatory importance in view of the forthcoming revision of the European Water Framework Directive. It is proposed that it should include the assessment of MP emissions into surface water and groundwater to ensure good qualitative and quantitative status of all European water bodies [32, 34]. But this requires the development of suitable research monitoring methodologies.

In previous projects, a methodology has been established using laboratory-scaled column percolation tests to assess the environmental compatibility of artificial turf and sports surface systems regarding the release of contaminants and their transport via the soil–groundwater transfer pathway [38, 43]. Results showed that it is necessary to take the entire structure of such systems into account, as there are no direct assessment criteria for the individual turf components. Such investigations were carried out for several common sports pitch installations. The contaminant concentrations in the eluates were compared with the limit values of a German regulation for the use of recycling materials in construction [5] which has been set into force in 2023. The concept for the risk assessment considering soil and groundwater protection has already been discussed [49]. According to the state of the art, up-flow column percolation tests under saturated conditions are used following standardised guidelines [17, 27, 30, 51]. So far, lysimeters using unsaturated conditions and somewhat larger dimensions have not yet been standardised, although they can provide a more realistic assessment of the leaching behaviour of contaminated soils. Laboratory lysimeters are usually irrigated from the top, which is closer to field conditions. The obtained eluates are subsequently analysed for organic and inorganic contaminants. Since MP science is a relatively new research field, to date, there are no standardised protocols for MP sampling from leaching tests to analyse and evaluate the transfer behaviour of MP through soil. Furthermore, classical lysimeters are i) not suitable because components can be made of target-polymers and thus do not fit the contaminant-specific material requirements (risk of cross-contamination); and ii) not made for MP sampling during long-term experiments. The main reason is that standard lysimeters do not have accessible filters that could allow for MP sampling. The only option would be an additional manual eluate filtering step of the large eluate glass collecting bottle, which can weigh up to 5 kg when empty. Consequently, the full heavy eluate collection bottles (total weight with sample: up to 25 kg) would have to be manually lifted for filtering and multiple rinsing, which is physically difficult to impossible and time-consuming. Additionally, this can pose high risks of injuries and cross-contamination when sampling for MP and dissolved contaminants.

To close this gap, we developed, constructed and in-house manufactured an innovative microplastics eluate lysimeter (MEL) which combines the simultaneous sampling of MP and released contaminants in one integrative laboratory lysimeter system to assess emissions from seepage water into groundwater layers or drainage water. The main objectives of the MEL construction were firstly to develop an automated, straightforward and easy-to-use system, which requires minimum maintenance for time-efficient long-term tests and could therefore be used for routine monitoring. Secondly, it had to meet all contaminant-specific material requirements: for MP detection the use of plastic-free materials or materials from non-target polymers, for heavy metals the non-use of brass components, and for PAH the use of polytetrafluoroethylene (PTFE) tubes. Finally, the system had to be safe and robust to generate reliable data regarding representativeness and reproducibility. Therefore, the MEL is based on a classic lysimeter setup, which has been upgraded and modified by the integration of a novel MP filter module. The module allows the direct filtration of the eluate through stainless-steel MP filter crucibles made with a geometric pore size of 5 µm [7, 8]. Ahead of the MP filter module, a stainless-steel sieve is installed to retain the soil sample (mesh size: 1,000 µm). Therefore, the MEL allows MP sampling of the health-relevant particle sizes of $1{,}000 \geq 5$ µm. The MP filter crucibles can be directly measured by thermal extraction desorption-gas chromatography/mass spectrometry (TED-GC/MS) without sample transfer losses and thus

reduced cross-contamination risks. TED-GC/MS is a fast automated method for the determination of MP masses, which uses polymer-specific decomposition products for MP detection [25, 26, 41]. Since its first introduction in 2015, TED-GC/MS established itself as an analytical MP monitoring method (ISO/DIS 16094–3: 2024, ISO/TR 21960: 2020), which provides robust data for various environmental matrices [9, 42, 53].

The key objective of this study was to evaluate the contaminant releases from artificial turf systems via seepage water by developing and optimising the MEL to obtain reliable and reproducible data. Therefore, this study compared environmental contaminant emissions of three artificial turf scenarios at different ageing states (fabric-new/unaged, artificially aged and real-time aged): i) the past (oTurf: old, fossil-based turf with synthetic infill), ii) the present (fTurf: most commonly installed turf in Germany, fossil-based with EPDM infill) and iii) the future (rTurf: turf with recycled grass fibres and no synthetic rubber infill). To simulate the outdoor stress during the turf lifespan of approximately 15 years, fabric-new turfs and EPDM granulate were accelerated aged by UV weathering and subsequent mechanical stress. Complementary to TED-GC/MS analyses for MP masses, the eluates were also analysed for PAH using gas chromatography/mass spectrometry (GC/MS) and for heavy metals using inductively coupled plasma-optical emission spectroscopy (ICP-OES).

Methods

Materials

Artificial turf components

Artificial turf pitch components were fabric-new and provided by Polytex Sportbeläge Produktions-GmbH (Grefrath, Germany), concretely rTurf (turf with recycled polyethylene (PE) grass fibres, LT Cross R 235 18/8), fTurf (fossil-based turf, LT Cross 235 18/8), the elastic layer and EPDM and sand infill. Figure 1a gives an exemplary schematic overview of an artificial turf system. rTurf and fTurf consisted of PE grass fibres which are tufted with winding yarn of polyethylene terephthalate (PET) into a geotextile or carpet backing of polypropylene (PP), which is glued on the back with polyurethane (PU) to secure the grass fibres (Fig. 1b). Additionally, sand infill is used to weigh down the artificial turf and to keep the grass fibres upright. For fTurf, additional EPDM granules were added (4.2 kg/m^2, Fig. 1c).

Underneath the artificial turf is an elastic layer mostly consisting of SBR granules from end-of-life car tyres bound with PU for better performance. Finally, an unbound base layer of mineral aggregates functions as a drainage layer and as a frost protection layer. The two analysed real-time aged old turfs (oTurf^1/oTurf^2) including their respective sand/synthetic infill mixtures, were provided by the artificial turf recycling company FormaTurf (Essen, Germany). oTurf^1 consisted of PE grass fibres, PP backing and PET winding yarn, a polystyrene-based glue and an EPDM/sand infill mixture. oTurf^2 was composed of PE grass fibres, PP backing and winding yarn, SBR-based glue and an unspecified thermoplastic elastomer (TPE) and sand infill mixture. All synthetic rubber/infill mixtures were representatively divided using a stainless-steel riffle splitter. All turf samples were first cut to pieces 20 cm in diameter and then nine small holes were randomly added into the backing to allow drainage of the eluent.

Unbound base layer

100 kg quartz gravel of different grain size fractions was washed and homogenised using an Eirich intensive mixer to obtain a grain size distribution in accordance with requirements for building material mixtures for frost protection layers [20, 35]. Thereby, the quartz fractions were mixed as follows: 2–4 mm (45%), 4–8 mm (45%) and 8–16 mm (10%). The gravel was then sieved to separate the fine fraction (< 2 mm), which was generated during mixing. Next, the gravel was divided into representative 25 kg portions and annealed overnight at 600 °C in ceramic trays to remove any potential plastic particles. After cooling down, the gravel was washed several times with deionised water until the water was clear to remove fine particles < 1 mm and then dried at 30 °C. Each MEL was filled with 10 kg gravel and irrigated for 7 h to remove the last fine adhesive gravel particles, which could potentially clog the MP filter crucibles. Stainless-steel round filters (mesh size: 5 µm, Gebr. Kufferath AG, Düren, Germany) were inserted into the MP filter module to remove larger gravel particles, which could potentially clog the small PTFE tubes. The gravel was then left in the MEL for approx. 2 d to allow residual water to run off. As the gravel was still moist, it was necessary to determine the saturation time using a complete test system to prevent residual water from the gravel being collected within the first fraction. The saturation time was 1 h.

Accelerated ageing of artificial turf

To simulate the outdoor weathering during the turf service lifespan of 15 years, the artificial turfs (rTurf/fTurf) and EPDM infill were accelerated aged using a combination of UV weathering and mechanical stress. First, the samples were UV irradiated in a weathering device (Global UV Test 200, Weiss Umwelttechnik GmbH, Reiskirchen, DE) using UVA-340 nm fluorescent lamps in accordance with DIN EN ISO 4892–3:2016 [22]. The temperature range was −10 to 70 °C, simulating both cold and warm exposure or summer and winter phases. A

24 h cycle was as follows: 1:13 h heating up, 21:04 h constant 70 °C, 1:13 h cooling down, 0:30 h constant −10 °C. The long 70 °C phases served as a temperature-induced reaction accelerator for polymer ageing. The artificial turf samples (dimensions: 80×40 cm) were vertically attached in the weathering device with metal hooks. EPDM granules were UV weathered horizontally with deflection mirrors in nine stainless-steel containers with sample weights of 150 g each (total: 1.35 kg). The artificial turf could not be subjected to irrigation or wet exposure, because potentially formed MP would have been flushed into the UV weathering device. As a compromise, a constant relative humidity of 90% was chosen. Since the EPDM granules were UV weathered horizontally, they were additionally exposed to moisture by daily manual watering of 100 mL of deionised water for each container. For the calculation of the irradiation time, the target irradiation of 15 years in Potsdam (2,700 MJ/m^2 UV, Deutscher Wetterdienst [15]) was used as an example for central German UV conditions. Thus, the artificial turf was UV irradiated for 16 d and the EPDM granules for 35 d. Further details on the UV irradiation time calculation can be found in Kittner et al. [40]. After UV weathering, the mechanical stress on artificial turf and its EPDM granules over its service lifespan of 15 years was simulated in a Lisport wear device (Labosport, Le Mans, FR) in accordance with DIN EN 15306: 2014 and FIFA guidelines [36]. For this purpose, the samples were subjected to 60,000 cycles with two stainless-steel rollers with polyamide cleats, each weighing 28.5 kg, whereby the second roller rotated at a 40% lower speed than the first roller. The sand and EPDM infill masses varied depending on the artificial turf type and were based on the manufacturer installation specifications for real sports pitches. Therefore, rTurf was mechanically stressed using 24.0 kg/m^2 sand, while for fTurf a mixture of 15.3 kg/m^2 sand and 4.2 kg/m^2 EPDM was used.

Construction of the microplastics eluate lysimeter

The MEL consists of three main components (Fig. 2a + b): 1. control module with irrigation system; 2. glass lysimeter with MP filter module and 3. eluate collecting bottles with vacuum coupling. Figure 2d shows the control module (BMT Fluid Control Solutions GmbH, Friedrichsdorf, DE), which is connected to the in-house deionised water supply (pressure: 5–6 bar). First, a water detector with an upstream solenoid shut-off valve is installed and the water detector placed on the laboratory floor as a safety measure to shut off the water supply in the event of a leakage. Subsequently, a water pressure regulator reduces the in-house line pressure to a water pressure of 1 bar for optimal irrigation of the mist spray nozzle. Following is a digital timer with a solenoid valve which allows automatic long-term experiments with variable irrigation intervals. The control module is connected via silicon hose with the irrigation system (Fig. 2e). There, the silicon hose is attached to a height-adjustable rod into which a nozzle head (spray angle: 80°, full cone spray pattern, Micro Rain Systems, Altenburg, DE) is screwed. The height adjustment enables different irrigation angles and sample filling heights. The irrigation characteristics can be changed by using different nozzle heads. This allows the simulation of fine mist, drizzle or rain. The rod is screwed into the lysimeter lid, which sits in the upper flange on top of the glass lysimeter (outer/inner diameter: 215/200 mm, height: 650 mm), which is firmly mounted to a table. The upper lid, upper and lower flange, rod and nozzle head are made of the non-target polymer polyvinyl chloride. The transparent glass cylinder allows the monitoring of the correct functioning of the MEL system and the general experiment process. The sample configuration corresponded to the schematic structure in Fig. 1a up to the building ground (Fig. 2f). The cylinder is placed on the lower flange (Fig. 2b + g), which is funnel-shaped for improved eluate run off.

On top of the lower flange is a stainless-steel sieve that retains large solid particles but allows MP (< 1 mm) to pass through. Following next is the MP filter module (Fig. 2h), which is newly constructed to automatically filtrate the eluate through MP filter crucibles (Fig. 2i, Gebr. Kufferath AG) and contain MP ≥ 5 µm, but can also be used with round filters. Finally, the eluate is collected through a PTFE hose in a 20-L glass bottle coupled with a vacuum pump that generates a low vacuum (700 mbar, Fig. 2a) to facilitate filtration through the small-mesh MP filter crucibles. Figure 2g shows an additional safety overflow (height: 200 mm) with a built-in sieve (mesh size: 5 µm) so that—in the event of MP filter crucible clogging—the eluate can be collected in a 10-L safety glass bottle. Both eluate collecting bottles are placed on a rolling board to transfer the bottles to a water pump and scale for i) collecting 1 L eluate samples for subsequent PAH and heavy metal analyses, ii) weighing the eluate for determination of the flow rate and liquid-to-solid ratio (l/s) of the fractions and iii) emptying the collecting bottles. For easy and fast emptying of the MEL after the experiments, a stainless-steel auxiliary device has been manufactured (Fig. 2c), which is secured around the MEL with a tension belt.

Simultaneous sampling concept

To unite the specific analysis requirements of the different environmental contaminants, a joint sampling concept was developed based on a similar to DIN 19528–23: 2023 [17] (up-flow laboratory-scaled column percolation test). The l/s ratios were determined using the total

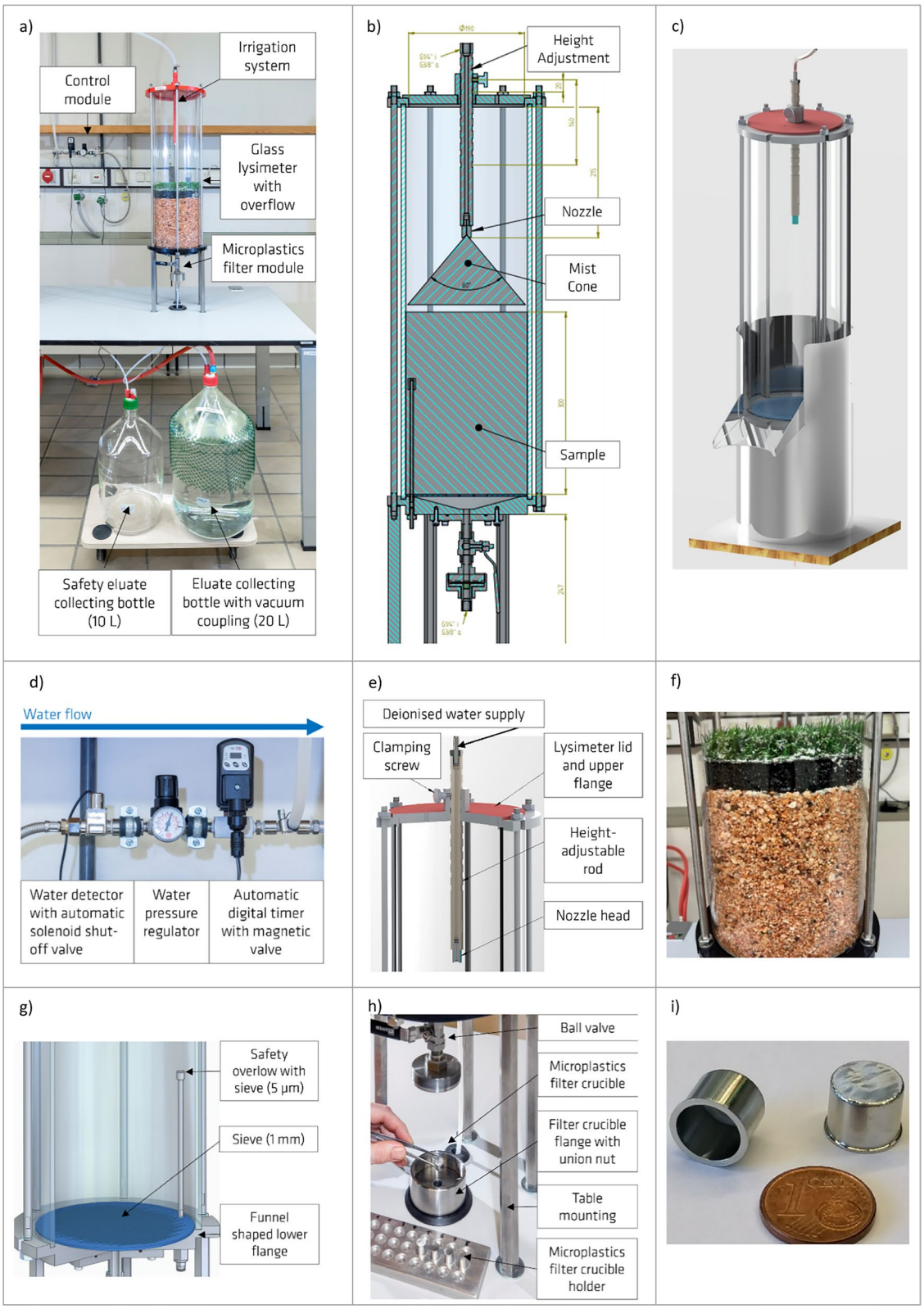

Fig. 2 Structure and main elements of the MEL: **a**) overview, **b**) technical drawing, **c**) auxiliary emptying device, **d**) control module, **e**) irrigation system, **f**) artificial turf system, **g**) lower flange with overflow, **h**) MP filter module, **i**) MP filter crucibles

sample masses (dry matter) and eluent volume resulting from the flow rate and percolation time (without saturation time). The targeted l/s of the fractions were modified and ranged from l/s 0.3 to 25. The total eluent volume was calculated corresponding to field conditions of the natural precipitation in Central Germany of an artificial turf sports pitch over its service lifespan of 15 years. Here, Potsdam served as a regional benchmark with an average annual precipitation of 585.8 L/m^2 [16]. Based on the glass cylinder area of 0.03 m^2, this corresponds to 18.4 L/year or a total target irrigation volume of 276 L for the simulation of 15 years. For time efficiency of the long-term experiments, in total four MEL were constructed and sampled simultaneously. To compare the effects of accelerated ageing on contaminant emissions, the MEL experiments were carried out with both farbric-new and accelerated aged artificial turf of the same sample (rTurf/fTurf), each over the same irrigation volume of 276 L. The eluent flow rate was measured twice: i) before the start of the experiment by collecting and weighing the obtained eluate over a defined time period and ii) continuously during the experiment by weighing the eluate volume obtained from the individual fractions. The irrigation time of the individual fractions was controlled by an automated time switch. To prevent clogging of the MP filter crucibles (Fig. 2f), the irrigation intervals were adapted to the expected MP emissions and were either set to 7 h or 3 h. At a flow rate of approx. 45 mL/min, this corresponded to eluate volumes of approx. 18 L or 8 L, respectively. Consequently, the number of fractions analysed varied between 18 and 35 microfilter crucibles per experiment.

For the determination of the solid matter content, the densities of the individual components were measured in accordance with DIN 66137–2:2023 [18] using a gas pycnometer (Ultrapyc 5000, Anton Paar, Ostfildern-Scharnhausen, DE) under a helium atmosphere at 20 °C. The density of the complete system was calculated as the mean value of the densities of the individual components, taking into account the mass fractions of the individual components in the complete system. Additionally, the total density of the complete system is required for further calculations of the flow regime parameters, e.g. pore volume, flow rate and contact time. The detailed data on the individual turf components of the column packaging can be found in the Supplementary Information (SI) in SI-Tab. 1.

Heavy metal analysis

The eluates were analysed for heavy metals using ICP-OES (iCAP7400, Thermo Fisher Scientific, DE, USA) in accordance with the current standards DIN EN ISO 22036: 2022 [23] and EN ISO 11885: 2009 [28]. Aliquots of 20 mL were taken from the eluates and preserved with five drops of 65% nitric acid based on DIN EN ISO 5667–3: 2018 [29] (pH < 2). Analyses were performed using the QTEGRA software (Thermo Fisher Scientific). The limits of quantification (LOQ) were determined in accordance with DIN 32645: 2008 [19], Formula 14, and can be found in SI-Tab. 2.

PAH analysis

PAH is a class of substances comprising several hundred individual compounds, which consist of interconnected aromatic benzene ring systems. For this assessment, 16 substances selected by the US EPA are analysed and summed up (^{16}PAH), which cover the spectrum from naphthalene with two aromatic rings to the higher molecular PAH up to six rings. The limit values were set for an l/s of 2 L/kg which has been proven to be suitable for risk assessment. PAH concentrations in the eluates were determined in accordance with DIN EN 17503: 2022 [21] using GC/MS. For sample preparation, 900 mL eluate was weighed in a 1-L DURAN® glass bottle and spiked with 100 µL internal standard (PAH-Mix 31, Dr Ehrenstorfer GmbH, Augsburg, DE, diluted with acetonitrile to a concentration of 1 mg/L each of naphthalene-d8, acenaphthene-d10, phenantrene-d10, chrysene-d12 and perylene-d12). 50 mL hexane was added and the mixture agitated in a horizontal shaker for 1 h at 125 rpm. The content of the glass bottle was then transferred to a 1 L separatory funnel by rinsing with ultrapure water and, after sufficient separation, the aqueous phase was isolated from the organic phase. The latter was transferred to a 200-mL Erlenmeyer flask and dried using sodium sulphate (Na_2SO_4) for a minimum of 30 min. The dry extract was then transferred to a 450 mL special vessel and residual Na_2SO_4 was washed three times with 20 mL hexane. The wash solution was combined with the extract in the Rocket vessel. To this mixture, 50 µL iso-octane was added as a keeper. The extract was then concentrated to approx. 200 µL using an evaporator (Rocket Synergy, Genevac Ltd., Ipswich, UK) with the "MTBE 100" method (35 °C, ΔT, final stage 2 min, cooler: −10 °C). The concentrated extract was quantitatively transferred to the volumetric flask and diluted up to 1 mL with hexane. Subsequently, the PAH in the extract were analysed using a GC/MS system with automatic autosampler (6890N, 7683B, Agilent, Santa Clara, CA, USA). The compounds were firstly separated in a chromatographic column (ZB-PAH-EU, Zebron, 2 min 50 °C, heating rate: 30 °C/min to 120 °C, 5 °C/min to 320 °C 6 min hold, helium flow: 0.8 mL/min) and then analysed in a mass spectrometer (ion source: 230 °C, quadrupole: 150 °C, electron impact ionisation: 70 eV, 5973, Agilent). The injection volume was 1 µL and the injector temperature 280 °C. The LOQ

was also determined in accordance with DIN 32645: 2008, Formula 14 [19] (see SI-Tab. 3).

Microplastics analysis

After sampling, the MP filter crucibles were oven-dried at 40 °C and weighed. MP masses were identified by TED-GC/MS using polymer-specific thermal degradation products and their specific retention times (t_R) and characteristic fragment ions. First, the crucibles were pyrolysed from 200 to 500 °C at a heating rate of 10 K/min using a thermogravimetric analyser (TGA2, Mettler Toledo Columbus, OH, USA, nitrogen atmosphere, flow rate: 50 mL/min). The operating temperature was optimised to the narrower temperature range of 200–500 °C (in previous work: 25–600 °C), as this is the range in which most polymers pyrolyse. As a result, only the thermal degradation products of the relevant temperature range are sampled, the solid-phase adsorber gets less loaded with irrelevant thermal degradation products (e.g. of environmental matrices) and the TED-GC/MS analysis is faster. Further details on the new methodological optimisations of the TED-GC/MS analysis will be soon available in Wiesner et al. (tba) [52]. The GC/MS measurements of the solid-phase adsorber (polydimethylsiloxane adsorber, Envea GmbH, Karlsfeld, DE) were performed in a gas chromatograph (7890, 5977B, Agilent), which allowed the analysis of the mass-to-charge ratio (m/z) from 35 to 350. Further information on the TED-GC/MS measurement principles and parameters is described in detail in the literature [1, 25, 41]. Within this work, the TED-GC/MS polymer marker pool was expanded and new polymer marker compounds for EPDM and PU were determined. An overview of all polymer marker compounds used for MP detection is given in SI Tab. 4. The polymer-specific limits of detection (LOD) and LOQ were determined from the fivefold or tenfold signal-to-noise ratio using the polymer marker compound and its m/z used for quantification and the enhanced ChemStation software (version 2015, Agilent). The determined LOD's were 1.1 µg for PE, 0.03 µg for PP, 0.2 µg for PET, 0.05 µg for SBR, 2.1 µg for EPDM and 0.3 µg for PU. For MP quantification, the polymer-specific response factors were determined by one-point calibrations of the respective turf system components. The LOQ's were as follows: 2.2 µg for PE, 0.06 µg for PP, 0.4 µg for PET, 0.1 µg for SBR, 4.2 µg for EPDM and 0.6 µg for PU. All presented TED-GC/MS results are above LOQ.

Quality control and assurance

To reduce the risks of cross-contamination, the MEL fulfils all contaminant-specific material requirements (MP: plastic-free or non-target polymer materials, PAH: PTFE hosing, heavy metals: no brass materials). Further, all laboratory work was carried out under maximum plastic-free conditions, with minimal sample exposure times and plastic-free equipment (e.g. stainless-steel or glass). After the experiments, all parts of the MEL were dismantled and thoroughly cleaned. The glass cylinder and all glass collecting bottles were first washed with soap, then rinsed with deionised water, ethanol and finally with acetone. The smaller MEL components (1 mm sieve, overflow filter, MP filter module, MP filter crucibles) were also first washed with soap, subsequently rinsed with deionised water and then cleaned in an ultrasonic bath, first with ethanol and then with acetone for 15 min each. The upper part of the MP filter module containing the ball valve needed to be cleaned manually due to avoiding damage to the sealing rings. Additionally, the PTFE and silicone hoses were rinsed with ethanol. For quality assurance and to avoid possible carry-over and contamination, blank values were carried out by irrigating the empty, sample-free MEL for 7 h or 18 L and subsequent TED-GC/MS, GC/MS and ICP-OES analyses, each in duplicate. The TED-GC/MS blank results showed minimal MP contamination in one blank with 0.01 mg/m^2 PP and 0.01 mg/m^2 PET, whereas the second blank was MP-free. Additionally, analytical blank measurements were carried out for TED-GC/MS analyses before each sample measurement, which corresponds to the procedural blank measurements.

Further, an initial screening of MP recovery rates was performed using a polymer mixture of two certified MP reference materials of PET (BAM-P206) and PE (BAM-P210) as well as a PP reference material candidate. Details about their particle size distributions can be found in the respective data sheets and reports in the BAM webshop (https://webshop.bam.de). Duplicate determinations were carried out to simulate different magnitudes of MP emissions: one with 0.3 mg each and a second one with 1 mg each of PET, PE and PP. The polymer mixtures were evenly distributed on the metal sieve of an empty MEL and irrigated for 7 h. The average flow rate was 44 ± 3 mL/min, resulting in a total irrigation volume of 18 L. The filter crucibles were then oven-dried at 40 °C and subsequently analysed by TED-GC/MS.

Results and discussion

Heavy metal emissions

The release of heavy metals from seepage water from artificial turf has been reported in previous studies [6, 45]. The strongest limit values of the German regulation [5] to be kept at l/s 2 L/kg for soil materials are 210 µg/L for zinc, 41 µg/L for copper, 31 µg/L for nickel and 19 µg/L for chrome. For recycling materials (quality RC-1), the limit values are less stringent: 110 µg/L for

copper, 150 µg/L for chrome and no limits for zinc and nickel. Figure 3 illustrates the results for these heavy metals for all turf system scenarios investigated. It shows that at l/s 2 L/kg there were no exceedances of the strongest German limit values. Only the initial concentrations for nickel were somewhat higher, representing the easily mobile fraction (so-called first flush) of nickel.

This could be due to a surface wash-off, particularly in the case of the new turf containing recycled grass fibres (rTurf_new). The zinc concentrations of real-time aged turf (oTurf^{2}) were at a higher level (up to 123 µg/L) in comparison to the values for the other turf systems. This indicates that in earlier productions of artificial turf components, here most likely the TPE rubber infill, more zinc oxide was used, which serves to vulcanise rubber. This is further emphasised by the fact that no Zinc was found in the eluates of the new, unaged turf systems. Zinc is mainly released from old tyres that are processed into SBR granules and used in the elastic base layer. Due to the varying quality of tyres, the zinc concentrations also fluctuated greatly in previous studies [10, 37, 38]. Ageing did not influence the chrome release significantly. But for zinc copper and nickel, the ageing led to somewhat higher releases, as can be seen in Fig. 3. There were no critical concentrations measured for other metals in the eluates. All heavy metal emission results can be found in detail in SI-Tab. 2.

PAH release

Figure 4 illustrates the release of the sum of ^{16}PAH ($\sum$ ^{16}PAH) during the MEL experiments. Results showed that all PAH concentrations were below the legal limits of BMUV [5] for $\sum$ ^{16}PAH at l/s 2 L/kg, although naphthalene is not considered for the limit values of eluates due to its volatile character. For all turf systems investigated, a decrease in PAH release is observed with increasing l/s. The highest initial concentration with $\sum$ ^{16}PAH of 1.4 µg/L was found in the new turf containing recycled

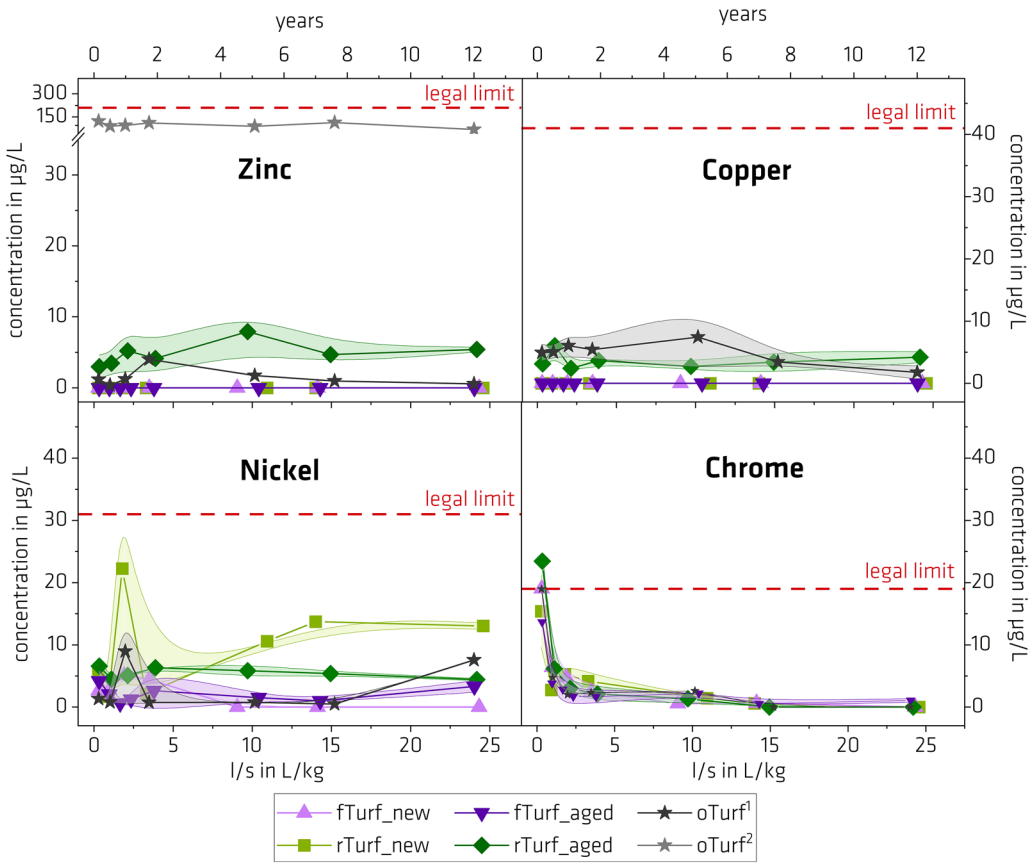

Fig. 3 Heavy Metals results of MEL experiments using ICP-OES. Shown are the mean metal concentrations with their ranges in µg/L with their ranges of the different ageing status of the analysed artificial turf scenarios over the course of the analysed liquid-to-solid ratio (l/s). As a rule of thumb, it can be assumed that an l/s of 2 L/kg represents roughly one year of irrigation under German conditions. Additionally, the red dashed lines present the respective German legal limits for soil materials at l/s 2 L/kg [5]. Since just the zinc concentrations of both real-time aged turfs (oTurf^{1}/oTurf^{2}) varied greatly, their zinc results are presented separately. For copper, nickel and chrome their values were similar and thus shown together

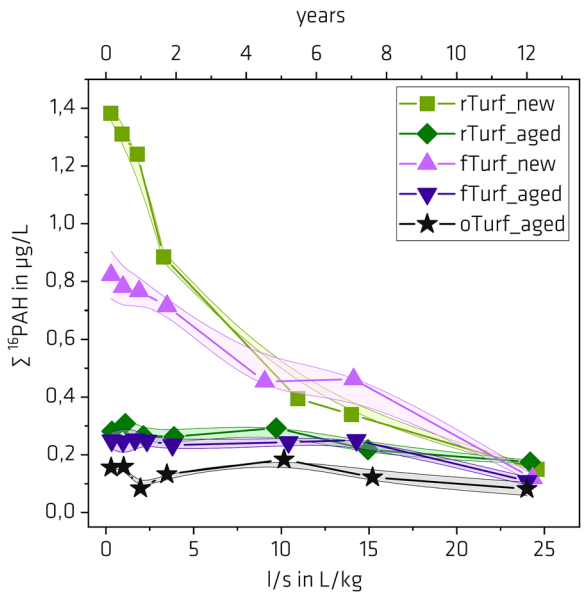

Fig. 4 PAH results of MEL experiments using GC/MS. Presented are the mean ∑ ^{16}PAH concentrations of all analysed artificial turfs systems at different ageing status with their ranges in µg/L over the course of the liquid-to-solid ratio (l/s). As a rule of thumb, it can be assumed that an l/s of 2 L/kg represents roughly one year of irrigation under German conditions. Both real-time aged turf (oTurf^{1}/oTurf^{2}) ∑ ^{16}PAH results were similar and thus shown together

grass fibres (rTurf_new), followed by the fabric-new turf containing fossil-based fibres (fTurf_new) with 0.8 µg/L. The limit value for the best quality of recycling material (RC-1 according to BMUV [5]) is 4 µg/L for ∑ ^{15}PAH (without naphthalene). The main amount of PAH in the eluates consisted of naphthalene, especially for the fabric-new systems (see Tab. SI–3). Additionally, acenaphthene, which is used in polymer production, is present in measurable concentrations, although its water solubility is relatively low. At the final l/s of the experiments (25 L/kg), the concentration of ∑ ^{16}PAH decreased to a comparable low level between 0.1 and 0.2 µg/L for all turf systems investigated, since the PAH with better water solubility are very likely almost depleted. It can be expected that the PAH release remains at a similar level in the following due to the very low water solubility of the substances with molecules of a larger size (higher number of aromatic rings). Ageing of turf components caused a lower release of PAH, as a loss of PAH occurred in the course of ageing. The concentration levels in the fabric-new systems can be considered maximum values for PAH emissions. For soil material, there is a limit value for benzo-a-pyrene of 0.2 µ/L available [5], which refers to the most cancerogenic PAH and was always kept for the eluates of all analysed artificial turf systems at l/s 2 L/kg.

Microplastics emissions

Recovery rates

The MP recovery rates differed depending on the polymer type. The individual mean recovery rates with their ranges were as follows: 91.1 ± 31.8% for PE, 45.4 ± 2.7% for PP and 95.4 ± 15.9% for PET. There are multiple potential reasons for the partially low recovery rates and high deviations. Under-quantification could occur due to the filtering setup with the MP filter crucibles, as only MP ≥ 5 µm is retained. Furthermore, over-quantification could be due to the release of residual MP particles from previous experiments that could not be removed by cleaning of the MEL, as the recovery rate experiments were performed after the experiments with the turf samples. However, the different particle size distributions of the representative test materials probably had the greatest influence on the recovery rates. Here, D50 is a central parameter that describes the median particle size distribution or the equivalent particle diameter of the measured volume, below which 50.3% of the particles lie [3]. Complementary analyses showed the following D50 values of the polymer test materials: 18.0 ± 0.2 µm for PE (BAM-P210, n = 30 measurements), 62.6 ± 1.9 µm for PET (BAM-P206, n = 30 [3]) and 261.7 ± 4.5 µm for PP (BAM-P208, n = 30). Therefore, PE had the lowest median particle size while having the highest recovery rate deviation and PP had the highest median particle sizes while having the lowest deviation. Since the recovery rate is a percentual value, the loss or gain of individual particles can have high influences on the recovery rate, which was particularly true for PP, which had the largest mean particle sizes. As a result, the surface tension of the water was probably too strong inside the MEL, and the relatively fine irrigation mist was too weak to transport the large and heavy PP particles to the filter crucibles.

Challenges of EPDM analysis using TED-GC/MS

For the quantification of MP emissions from synthetic rubber infill, the TED-GC/MS polymer marker pool had to be expanded to include EPDM detection. Using non-target analyses of different EPDM granules and a literature review [11, 50], a homologous series of triplets each consisting of methyl alkene, alkene and alkane were identified as the main thermal decomposition products. Since alkanes and alkenes are non-specific thermal decomposition products which can originate from different aliphatic compounds, seven methyl alkenes were chosen as characteristic polymer marker compounds for TED-GC/MS analysis (see SI-Tab. 4). For validation in soil matrices, two terrestrial middle earths (organic contents: < 1%) were spiked with two different EPDM granules at different weight percentages (wt%), in duplicate. The soil

sample masses were 20 mg and 50 mg and EPDM masses between 0.2 and 0.3 mg, corresponding to 0.5 wt% and 1 wt% EPDM. Although some polymer markers (EPDM-1, EPDM-2, EPDM-6) were difficult to detect due to coelutions with organic compounds from the soil matrix, the results showed overall a good identifiability of most EPDM markers (EPDM-3, EPDM-4, EPDM-5, EPDM-7) in both matrices at different concentrations. However, the recovery rates revealed that the quantifiability of EPDM in both soil matrices is affected by matrix compounds. As a result, at high EPDM concentrations (1 wt%), the uncertainties were lower with recovery rates of 67% and 100% than at low EPDM concentrations (0.5 wt%) with recovery rates of 163% and 243%.

In the next validation step, EPDM granules were analysed in a polymer matrix consisting of artificial turf components (PE, PP, PET, PU). The results showed a recovery rate of EPDM of 127 ± 8% and thus an over-quantification, indicating that the new EPDM markers were also present in other turf components. Detailed TED-GC/MS analyses showed that the EPDM markers were only found in the analysis of pure PE of the grass fibres, but not in the other turf components. Therefore, using methyl alkenes as EPDM markers could potentially lead to EPDM over-quantification when PE particles are present in the sample. The similarity of the thermal decomposition products of EPDM and PE can be explained by their polymeric molecular structures, as both contain ethylene units. Since EPDM also consists of a propylene and a diene unit, here 5-ethylidene-2-norbonene according to the manufacturer, less dominant thermal degradation products were further examined to overcome this analytical challenge. Unfortunately, this approach was not successful because either the EPDM thermal pyrolysis degradation products described by other researchers, e.g. 3- and 4-ethylidene-1-cyclopentene [11], were not detectable or non-specific. Another approach was indirect detection via EPDM vulcanisation agents, e.g. benzothiazole or 2-methyl-benzothiazole. However, detailed analyses of TED-GC/MS results showed that this was unsuitable due to high detection inconsistencies and thus posed the risk of EPDM under-quantification.

In conclusion, the analytical challenges of EPDM detection using TED-GC/MS analysis have not yet been resolved and both analytical approaches can lead to either potential over- or under-quantification of EPDM mass contents in unknown samples. Due to a lack of alternatives and because risk assessment is of crucial importance within this project, the decision was made to use the seven methyl alkenes listed in SI-Tab. 4, knowing well that this could lead to a potential over-determination. For this reason, the best identifiable thermal decomposition product, 2-methyl-1-undecene (EPDM-3), was used for EPDM quantification to estimate maximum MP emissions of EPDM rubber infill.

Microplastics masses

Figure 5 shows the summed total mean MP results (log) over the total irrigation volume, which can be found in detail in Table 1. The highest MP contents were detected in both real-time aged oTurf[1]/oTurf[2] representing the past scenario (∑MP: 136.4–252.5 mg/m^2). The main MP emissions sources were the synthetic rubber infills (∑EPDM/TPE: 42.2–192.5 mg/m^2) and the grass fibres (∑PE: 21.7–38.7 mg/m^2). This can be explained by the fact that on an artificial turf, outdoor stress caused by weathering (e.g. by UV radiation or photo-oxidation) and mechanical stress (e.g. by sand infill, studs on football shoes, artificial turf maintenance machines) mainly affect the rubber granules and the grass fibres, leading to material fragmentation. In this context, it was surprising that only comparably low PP contents formed by the backing surfaces were detected in both real-time aged oTurfs (∑PP: 1.9–2.3 mg/m^2), since they were also exposed to real outdoor stress. This indicates that the backing is protected by the rubber granules against the outdoor stress, especially from the abrasion effects caused by the sand infill.

Additionally, high contents of the backing glue were detected in oTurf[1] with 69.6 mg/m^2 and oTurf[2] with 19.5 mg/m^2. This is probably due to the sample placement in the MEL glass cylinder, as i) the backing glue was

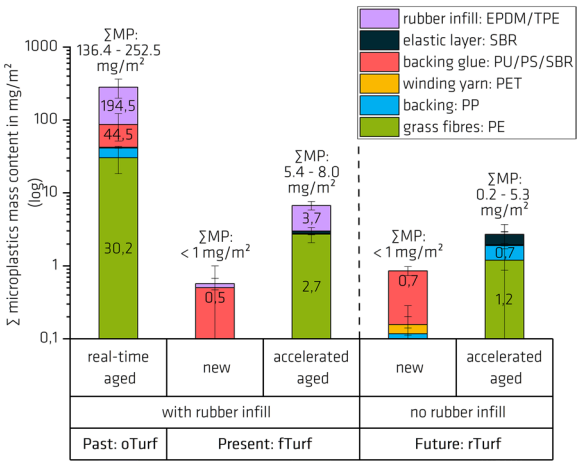

Fig. 5 MP mass results of MEL experiments using TED-GC/MS. Shown are the mean ∑MP masses in mg/m^2 with their ranges (log), subdivided in artificial turf scenarios with and without synthetic rubber infill and their ageing status. The polymer abbreviations are as follows: polyethylene (PE), polypropylene (PP), polyethylene terephthalate (PET) polyurethane (PU), polystyrene (PS), styrene butadiene rubber (SBR), ethylene propylene diene monomer rubber (EPDM), thermoplastic elastomer (TPE)

Table 1 Summary of MP emissions of all analysed artificial turf scenarios of different ageing states expressed in mg/m^2

Polymer type	Artificial turf component	Microplastics masses of artificial turf scenarios in mg/m^2					
		Past: oTurf		Present: fTurf		Future: rTurf	
		Real-time aged oTurf [1]	Real-time aged oTurf [2]	Fabric-new	Accelerated aged	Fabric-new	Accelerated aged
EPDM	Rubber infill	42.15	–	0–0.15	3.06–4.32	–	–
TPE	Rubber infill	–	192.54	–	–	–	–
PE	Grass fibres	21.70	38.65	–	2.31–3.18	0–0.26	0–2.39
PP	Backing	2.28	1.89	–	0.04–0.05	0–0.03	0.01–1.46
PET	Winding yarn	–	–	–	–	0.01–0.07	0–0.11
SBR	Elastic layer/backing glue	0.67	19.47	–	0–0.48	–	0–1.42
PS	Backing glue	69.58	–	–	–	–	–
PU	Backing glue	–	–	0.13–0.79	–	0.58–0.76	–
∑MP in mg/m^2		**136.38**	**252.54**	**0.13–0.94**	**5.41–8.02**	**0.83–0.89**	**0.16–5.28**

Since duplicate determinations were carried out, the polymer mass contents are shown with their value ranges. A hyphen indicates that no MP content above the limit of quantification was detected

already brittle and ii) cutting out the round 20 cm turf samples could have led to fraying of the edges and thus to increased MP formation. Further, it is important to note that the backing glue of oTurf[2] was SBR-based and had therefore the same thermal decomposition products as SBR, making them analytically indistinguishable from the SBR elastic layer. However, since the backing glue was so brittle, it can be assumed that the majority of the detected SBR can be assigned to MP emissions from the backing glue.

The TED-GC/MS results of the present scenario showed that for the fabric-new fTurf, minimal total MP emissions (∑MP: 0.1–0.9 mg/m^2). This was mainly from the PU backing glue, probably due to residual particles produced when cutting the turf samples. Additionally, only minimal emissions from the rubber infill (∑EPDM: 0–0.2 mg/m^2) were detected. These were probably smaller particles formed during production or transport, as the granules are produced with a target size > 1 mm (Fig. 1c). The accelerated ageing of fTurf led to MP increases of approximately tenfold and thus to total MP emissions of 5.4–8.0 mg/m^2, mainly from the rubber infill (∑EPDM: 3.1–4.3 mg/m^2) and grass fibres (∑PE: 2.3–3.2 mg/m^2). Further, minor emissions of the PP backing and SBR elastic layer were detected.

The future scenario had overall the lowest MP emissions of all analysed turf scenarios. Like the present scenario, the fabric-new rTurf had only minor total MP emissions (∑MP: 0.8–0.9 mg/m^2), mainly from the backing glue (∑PU: 0.6–0.8 mg/m^2) which was probably due to particle residues formed during the sample cutting. After accelerated ageing, the MP emissions also increased by approximately a fivefold to a total MP content of 0.2–5.3 mg/m^2. Here, the main MP emission rTurf components were the grass fibres (∑PE: 0–2.4 mg/m^2), backing (∑PP: 0–1.5 mg/m^2) and the elastic layer (∑SBR: 0–1.4 mg/m^2). For comparison, e.g. with field data, all MP results are also available in SI-Tab. 4 expressed in the unit µg/L. The MP contents in µg/L differ marginally from those in mg/m^2, as the irrigation volume per MEL differed slightly per experiment.

Microplastics emissions per artificial turf sports pitch

All MP results are expressed in the unit mg/m^2 to form a database that enables an extrapolation of MP emissions per artificial turf pitch of any size. The aim here is to get an idea of the approximate order of magnitude of maximum MP emissions. When extrapolating the MP results for an aged standard artificial turf sports pitch (7,000 m^2) during its service lifespan of 15 years, the MP emissions from seepage water into the groundwater layers or drainage water correlated to 954.7–1,767.8 g for the past, 3.8–56.1 g for the present, and 1.1–37.0 g for the future scenario. To our best knowledge, this is the first published data on experimentally determined mass content-based results of MP emissions from seepage water of artificial turf sports pitches. Overall, the MEL results are indicating that MP emissions towards the groundwater layers or drainage water are comparably low, which confirms the theoretical evaluation of Bertling et al. [4]. However, all MP emission paths must be included in a total MP assessment, e.g. wind, water run-off and artificial turf maintenance.

Conclusions

Although there is data on the release of pollutants for individual components of artificial turf systems, it is not possible to derive an assessment of the risk to soil

and groundwater from this. Instead, the approach of considering the entire structure of artificial turf systems has once again proven its worth in this project since it was also suitable for quantifying the discharge of MP. The artificial turf results showed that all contaminant emissions of PAH and heavy metals were under the German legal limits and thus uncritical—also for the aged samples. Regarding the MP emissions, all fabric-new turf showed minimal MP emissions, while turf ageing led to MP increases, mainly from synthetic rubber infill and grass fibres. In practice, the newly developed MEL proved to be a suitable holistic sampling device for simple, straightforward and automated monitoring of particulate and dissolved contaminant emissions from simple soil matrices, like artificial turf, from seepage water into the groundwater layers. Additionally, the MEL could also be used for emission analyses of other materials, e.g. for building or construction, to investigate MP transports and potential leaching of hazardous substances. In conclusion, the MEL has the potential for implementation in future research concepts and may be applied to other matrices to obtain data in the context of the European Water Framework Directive and future European regulations.

Abbreviations

EPDM	Ethylene propylene diene monomer rubber
fTurf	Artificial turf with fossil-based PE fibres with EPDM rubber infill representing the present scenario
GC/MS	Gas chromatography/mass spectrometry
ICP-OES	Inductively coupled plasma-optical emission spectrometry
l/s	Liquid-to-solid ratio
LOD	Limit of detection
LOQ	Limit of quantification
m/z	Mass-to-charge ratio
MEL	Microplastics eluate lysimeter
MP	Microplastics
oTurf	Old, real-time aged artificial turf with fossil-based PE fibres and synthetic rubber infill representing the past scenario:
oTurf[1]	With EPDM infill, oTurf[2] with TPE infill
PAH	Polycyclic aromatic hydrocarbons
PE	Polyethylene
PET	Polyethylene terephthalate
PP	Polypropylene
PTFE	Polytetrafluoroethylene
PU	Polyurethane
rTurf	Artificial turf with recycled PE fibres without rubber infill representing the future scenario
SBR	Styrene butadiene rubber
TED-GC/MS	Thermal extraction desorption-gas chromatography/mass spectrometry
TPE	Thermoplastic elastomer
t_R	Retention time
wt%	Weight percentage

Supplementary Information

The online version contains supplementary material available at https://doi.org/10.1186/s12302-025-01235-1.

Supplementary file 1

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Author contributions
MK supervised the project, curated the data, visualised the figures and wrote the original draft. All authors reviewed and edited the final manuscript. UK and KA acquired the funding, with UK being the project administration. UK, KA, MK, BC and SS conceptualised the study. The methodology was developed and validated by MK, BC, KA and UK. SS designed and engineered the Microplastics Eluate Lysimeter. The experimental investigations were carried out by MK, BC and TW. The data were formally analysed by MK and UK.

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Data availability
The datasets used and analysed during the current study are available from the corresponding author on reasonable request.

Declarations

Ethics approval and consent to participate
Not applicable.

Consent for publication
Not applicable.

Competing interests
The authors declare no competing interests.

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