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Release and quantification of organic and inorganic contaminants from geotextile materials in dynamic surface leaching test

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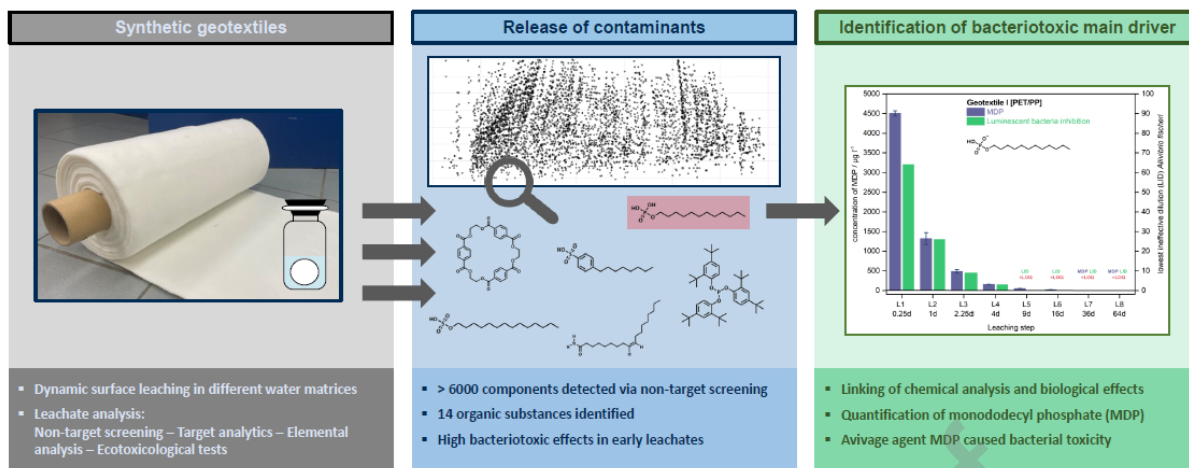
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ABSTRACT

Geotextiles are frequently used in waterway construction, for instance as filtration or separation layers of river banks and canals. A variety of additives are added to improve their lifespan during application and avivage agents are applied to optimize the manufacturing process. Two nonwoven needle-punched geotextiles were leached with deionized, ground and river water via the dynamic surface leaching test. Organic compounds released by the materials were identified via non-target screening using liquid chromatography quadrupole time-of-flight mass spectrometry (LC-QToF-MS). A variety of the identified compounds belong to the groups of sulfates, (benzene)sulfonates, polyethylene glycols, phosphates, carboxylic acids (amides), plastic-oligomers, antioxidants and light stabilizers. The luminescence inhibition of *Aliivibrio fischeri*, observed in leachates of tested polyethylene terephthalate/polypropylene (PET/PP) geotextile, can mainly be attributed to the released anionic surfactant monododecyl phosphate, that was identified by a combination of biotests and chemical analysis. This surfactant is obviously used as an avivage agent during geomembrane production. Target analysis via inductively coupled plasma mass spectrometry (ICP-MS) and liquid chromatography mass spectrometry (LC-MS/MS) ultimately elucidated that elements such as antimony and zinc as well as a multitude of organic substances are mainly released at the beginning probably by wash-off effects, while their release after the third leaching step was negligible.

Abbreviations: ATR-FTIR, attenuated total reflection fourier transform infrared spectroscopy; CE, collision energy; DOC, dissolved organic carbon; DSLT, dynamic surface leaching test; ESI, electrospray ionization; GW, groundwater; HALS, hindered amine light stabilizer; ICP-MS, inductively coupled plasma mass spectrometry; LC-MS/MS, liquid chromatography mass spectrometry; LID, lowest ineffective dilution; LC-QToF-MS, liquid chromatography quadrupole time-of-flight mass spectrometry; LOD, limit of detection; LOQ, limit of quantification; MDP, monododecyl phosphate; MRM, multiple reaction monitoring; MS¹, precursor ion; MS², daughter ion; NTS, non-target screening; PA, polyamide; PE, polyethylene; PET, polyethylene terephthalate; PEG, polyethylene glycol; PNEC, predicted no-effect concentration; PP, polypropylene; RT, retention time; RW, river water; SPE, solid phase extraction; YES, Yeast estrogen screen.

Graphical abstract



Keywords: Geotextile; Leaching; Non-target screening; Avivage agent; Ecotoxicity

Environmental Implication

Geotextiles include additives, which are used in manufacturing process. These chemicals can be released into the environment as potential contaminants after the material has been installed. The geomembrane is directly in contact with the surrounding water and has also an impact on the aquatic biota. Substance release into water phase has not yet been sufficiently investigated. Our study includes identification of released substances in different water matrices and their effects in ecotoxicological tests. We used a combination of chemical analysis and biotests to identify and quantify the main driver of toxicity to luminescent bacteria from a large number of compounds.

1. Introduction

Geotextiles consisting at least of one polymer belong to the group of geosynthetics. They have been used for the stabilization of canals, dikes, shores and river banks in waterway construction (Markiewicz et al., 2022). The majority of geotextiles are made from basis polymer fibers of the polyolefin family. Polypropylene (PP) is used approx. 90% in geotextiles followed by polyethylene (PE) with approx. 5%. The remaining 5% can be attributed to polyethylene terephthalate (PET) and polyamide (PA) (Hsuan, 2016; Wiewel and Lamoree, 2016). Woven, nonwoven and knitted geotextiles are known categories (Müller and Saathoff, 2015). They are thin, have a high strength, but are also flexible and water-permeable. Their geotechnical relevance in civil and environmental engineering has steadily increased since they first went on the market in around 1956 in the Netherlands (Koerner, 2016; Wiewel and Lamoree, 2016) and the first use of nonwoven geotextile in 1968 in France (Rawal et al., 2010). Compared to impermeable and composite materials, they are the most widespread product of geosynthetics (Koerner, 2016). With an annual growth rate of 11.9%, the global geotextiles market generated a revenue of 4.6 billion US dollars in 2019 (Wu et al., 2020). Geotextiles are able to carry elevated loads, separate and stabilize soil layers, prevent erosion, and drain and filter water (Bai et al., 2022). The materials have a high robustness against waves and hydraulic forces, elevated lifespans and good drapeability (Rawal et al., 2010). Their limited production and transportation costs are also advantages of geotextiles (Bezuijen and Vastenburg, 2013) compared to other filter materials used in hydraulic application such as sand, gravel or crushed stone of different grain and pore sizes (Heerten and Wittmann, 1985). These natural backfills require a large and heavy size of material and bank excavation with an effective distribution of material to obtain a strong protection. By contrast, the installation of the geosynthetic material is more flexible but needs to be covered with armor stones. The material comes into direct contact with soil, rocks, water bodies and other geotechnical materials after installation (Carneiro, J. et al., 2018). Geotextiles have been further developed in recent decades so that (bio)degradation is prevented by using additives and to ensure a longer lifetime (Hsuan, 2016). Polymers show good resistance to weathering, to chemical and biological degradation, while oxidation or prolonged contact with water might lead to alterations of the material by leaching of the additives (Carneiro, J.R. et al., 2018). Geotextile material potentially contains a large variety of additives such as hindered amine light stabilizers (HALS), UV stabilizer, antioxidants, flame retardants, processing chemicals, lubricants and avivage agents (Hsuan, 2016; Richter, 2009; Wiewel and Lamoree, 2016). Throughout manufacturing, process modifiers such as lubricants, smoothing agents, antistatic agents and other auxiliaries are used and applied to assist in the needling and bonding process of individual extruded plastic filaments into a nonwoven fabric (Bérubé and Saunier, 2016). These chemicals are distributed in the material after the manufacturing process and are often deposited as a thin film layer on the geotextiles. The manufacturer has to specify the base polymers, while the additives used are not reported. Hence the release of additives is largely unknown, as are their behavior and occurrence in the environment and their ecotoxicological relevance (Wiewel and Lamoree, 2016). Additive leaching and extraction methods from plastics are well known (Bridson et al., 2021). There are several studies combining leaching study with biotests (Bandow et al., 2018) for example with biocides from facades (Vermeirssen et al., 2018) and also in particular studies focusing on plastic products (Lithner et al., 2009; Sudar et al., 2013). The aqueous leachates can be investigated by chemical analysis and ecotoxicological tests for different construction materials (Bell et al., 2020; Gartiser et al., 2017b; Heisterkamp et al., 2023). Using effect-directed analysis of the different leaching fractions, the identification of currently unknown compounds is feasible (Tousova et al.,

2018). A variety of acute biotests has been recommended for aqueous samples such as assays with algae, daphnia, fish egg, estrogenicity and mutagenicity (Brack et al., 2019). Knowledge of the release and behavior of organic hazardous substances from construction materials is limited (Wangler et al., 2012). For this reason, dynamic surface leaching test (DSLTL) is a standardized lab-scale batch system to determine and evaluate the total release of all leachable substances from building materials into the water phase and suitable for investigation of released substances (Heisterkamp et al., 2021). DSLTL has been performed on a variety of building materials such as coatings, varnishes, plastic composites, grouts, granules and geosynthetics (Gartiser et al., 2017a; Heisterkamp et al., 2023). The leachates were analyzed for a variety of parameters such as dissolved organic carbon (DOC), selected elements, organic compounds and an ecotoxicological biotest battery. Scholz et al. (2021) provide DSLTL leaching linked to acute and chronic ecotoxicological tests (algae and daphnids) with artificially aged PP geotextile materials, resulting in no toxic effect. Deutschmann and Hollert (2020) and Scheres et al. (2022) investigated synthetic geogrid (PET) and nonwoven fabric (PP) with zebra fish egg (*Danio rerio*) test, which show no teratogenic effect, while lethal effects occurred when the leaching was prepared with Soxhlet extracts. They also point to the weak data situation with regard to the ecotoxicological assessment of geotextile leachates and released contaminants and recommend further biotests with different species and specific endpoints.

Our study focuses on the identification of compounds and elements that are released in contact with different water matrices from nonwoven monolayer geotextiles produced with the needle punch technique. As foreseen in DSLTL (CEN, 2014) we used deionized water. Standardized leaching tests are recommended for comparability (Heisterkamp et al., 2023). However, our study was extended to groundwater and river water to evaluate the transference of the DSLTL results with deionized water, which is a worst-case approach. Detection was performed for elements via inductively coupled plasma mass spectrometry (ICP-MS), for organic compounds via liquid chromatography mass spectrometry (LC-MS/MS) and via full non-target screening (NTS) using liquid chromatography quadrupole time-of-flight mass spectrometry (LC-QToF-MS). In combination with ecotoxicological tests, we focus on the identification of leached hazardous substances.

2. Material and methods

Geotextile materials were characterized by spectroscopy and were completely digested by microwave digestion followed by elemental analysis via ICP-MS to determine the maximum content of elements in the material. Leaching studies with geotextiles were carried out with a focus on dynamic surface leaching with deionized water (64 days) and shortened dynamic surface leaching (4 days) with groundwater and river water to investigate the leaching behavior of substances from the material into the aqueous matrices and to determine leachate toxicity. Leachates were analyzed for basic chemical parameters, elements, organic compounds identified in non-target screening and toxicity in biotests with different biological endpoints (luminescent bacteria inhibition, daphnia immobilization, algae growth inhibition, yeast estrogen screen, Ames). Selection of biotests was determined by their toxicity in leachates of a static surface leaching (28 days). Substance with a possible toxic effect were quantified via LC-MS/MS. Material used and the methods applied are described below in detail.

2.1. Geotextiles

Two different nonwoven geotextiles (categorized according to DIN (2018)) made of randomly orientated fibers were selected due to their frequent use in waterway construction as they are

regularly used as separation and filter elements in canals and rivers. They consist of white needle-punched and mechanically bonded polymer fibers. The selected materials differ in their polymer composition. The first nonwoven geotextile product labelled Geotextile I contains 70% PET and 30% PP (mass per unit area approx. 500 g m^{-2}) and the second product labelled Geotextile II is composed of 100% PP (mass per unit area approx. 800 g m^{-2}). Further specified product information according to the German technical delivery conditions for geotextiles and geotextile-related products on navigable waterways (BAW, 2021):

Geotextile I [PET/PP] for soil type A: Layer thickness: $> 4.5 \text{ mm}$; Tensile strength: longitudinal $\geq 12 \text{ kN/m}$, transverse 25 kN/m .

Geotextile II [PP] for soil type C: Layer thickness: $\geq 6.0 \text{ mm}$; Tensile strength: longitudinal $\geq 35 \text{ kN/m}$, transverse 55 kN/m .

Durability is declared for each product for 100 years in natural soils with $4 \leq \text{pH} \leq 9$ and soils temperature $\leq 25 \text{ }^\circ\text{C}$. Characterization of synthetic polymer material was performed via attenuated total reflection Fourier transform infrared spectroscopy (ATR-FTIR) from Frontier FT-IR spectrometer, PerkinElmer and a complete oxidative microwave digestion and following elemental analysis with ICP-MS (8800 Triple Quadrupol ICP-MS, Agilent Technologies). Details of the methods used are provided in SI Chapter S1 (ATR-FTIR) and SI Chapter S2 (digestion).

2.2. Chemicals

Deionized water (Milli-Q® IQ 7003 Pure and Ultrapure Water Purification System, Merck; $18.2 \text{ M}\Omega\cdot\text{cm}$ at $25 \text{ }^\circ\text{C}$; TOC: 1.8 ppb ; $0.22 \text{ }\mu\text{m}$ filter, Millipak) was used for DSLT and as the mobile phase for LC-MS/MS and LC-QToF analysis. Acetonitrile (LiChrosolv hypergrade, Merck) was used as the organic mobile phase. Sodium octylbenzene sulfonate (CAS: 6149-03-7), sodium dodecylbenzene sulfonate (CAS: 25155-30-0), sodium decane sulfonate (CAS: 13419-61-9), sodium tetradecyl sulfate (CAS: 1191-50-0), polyethylene glycols [PEG-400] (CAS: 25322-68-3), oleamide (CAS: 301-02-0) and Sodium monododecyl phosphate (CAS: 2627-35-2) were purchased from Sigma Aldrich as standards for identified compounds via NTS. Tri- (CAS: 112-27-6), tetra- (CAS: 112-60-7), penta- (CAS: 4792-15-8) hexaethylene glycol (CAS: 2615-15-8) were from Fisher Scientific. Ethylene terephthalate cyclic trimer (CAS: 7441-32-9) and -dimer (CAS: 24388-68-9) were from TRC and Tris(2,4-di-tert-butylphenyl) phosphite (CAS: 31570-04-4) was from Clariant (full list see SI Table S9).

2.3. Leaching study design and sample preparation

The release study was conducted according to DSLT guideline CEN/TS 16637-2 (CEN, 2014) for plate-like products. The leaching of the materials was carried out as triplicates. The individual leaching steps were performed at ambient temperature ($19\text{-}22 \text{ }^\circ\text{C}$) in deionized water to establish a worst-case release scenario and to fulfil the requirements of the DSLT. Two-disc shape pieces (diameter: 6.5 cm , one-sided surface: 66.4 cm^2 , mass used: Geotextile I [PET/PP]: approx. 3.4 g per sample; Geotextile II [PP]: approx. 4.6 g per sample) of both geotextile fleeces were placed in closed 250-ml amber glass bottles and covered with 150 ml of deionized water with a liquid to one surface area ratio L/A : 22.6 l m^{-2}). The sample pieces were cut with cleaned metal laboratory scissors to avoid contaminations. Temperature and humidity were constantly monitored and the bottles were placed in the dark. Closed glass bottles filled only with deionized water (without geotextile) served as negative controls. After sampling the aqueous leachates, they were stored in closed glass vessels at a constant temperature of $4 \pm 1 \text{ }^\circ\text{C}$. The physical, chemical and biological parameters were determined in the leachates according to the analytical methods described below. DSLT samples were taken at eight defined leaching steps (L) in the frame of 64 days in total, L1: 0.25 d , L2: 1 d , L3: 2.25 d , L4: 4 d ,

L5: 9 d, L6: 16 d, L7: 36 d and L8: 64 d. The leaching medium was completely renewed after each sampling. Temperature, pH and conductivity were measured with a multi-parameter measuring device (Multi 3630 IDS, WTW) directly after each leachate sampling. Dissolved organic carbon (DOC) was determined with DimaTOC 2000, Dimatec. DOC method according to (DIN, 2019) is described in SI Chapter S4 in detail. The release mechanism was calculated according to Annex B of CEN/TS 16637-2 (CEN, 2014).

For the testing of estrogenicity in Yeast estrogen screen (YES) and mutagenicity in Ames fluctuation test, samples were concentrated 1000-fold by solid phase extraction (SPE) using OASIS HLB 6 cc (200 mg) cartridges. The cartridges were conditioned with 2 ml n-heptane (Picograde, Promochem), 2 ml acetone (Picograde, Promochem), 3 x 2 ml methanol (Optigrade, Promochem) and 4 x 2 ml double distilled water and then loaded with 1000 ml of the aqueous samples. After drying, the SPE cartridges were eluted with 4 x 2 ml methanol. Extracts were evaporated, aliquoted and restored either in 800 ml DMSO (ReagentPlus, Sigma-Aldrich) or 200 ml ethanol (Optigrade, Promochem).

For the selection of sensitive bioassays, a preliminary leaching test was conducted to recognize toxic effects on tested species. A 28-day static surface leaching test with Geotextile I [PET/PP] and Geotextile II [PP] was performed with the same L/A and starting conditions described above. For that, geotextile material was placed in test bottles and covered with leaching medium but without changing the water during the leaching period. The results of biotesting on leachates is presented in Figure 3.

In order to compare the results obtained in contact with deionized water and the transferability of results to environmental waters, the DSLT was also performed with groundwater and river water. For this reason, leaching steps L1 (0.25 d) to L4 (4 d) were taken with Geotextile I [PET/PP] in contact with groundwater and river water as triplicates. The test was limited to four leaching steps (4 d). The groundwater was collected from a well in Koblenz-Arenberg, Germany (pH 7.2; conductivity: $546 \mu\text{S cm}^{-1}$) and is labelled [GW] in the following. River water was taken from the Rhine at river kilometer index 590.3, Germany (pH 8.0; conductivity: $455 \mu\text{S cm}^{-1}$) and is labelled [RW] in the following. A full list of sum parameters can be found in the SI (Table S10-1 and Table S10-2). Groundwater and river water without contact to geotextiles were tested as blanks.

2.4. Analytical methods

2.4.1. Elemental analysis

Unfiltered leachate and blank samples from the DSLT were filled in PP tubes (metal-free, VWR) and were mixed with 2% sub-boiled HNO_3 (Emsure 65%, Merck) for element analysis with ICP-MS (8800 Triple Quadrupole ICP-MS, Agilent Technologies). 28 elements were determined: aluminum (Al), silicon (Si), phosphorus (P), sulfur (S), titanium (Ti), vanadium (V), chromium (Cr), manganese (Mn), iron (Fe), cobalt (Co), nickel (Ni), copper (Cu), zinc (Zn), arsenic (As), selenium (Se), molybdenum (Mo), silver (Ag), cadmium (Cd), tin (Sn), antimony (Sb), thallium (Tl), lead (Pb), and uranium (U). In addition to oxidative microwave digestion, method of ICP-MS was expanded of sodium (Na), magnesium (Mg), potassium (K), calcium (Ca) and strontium (Sr). SLRS-6 (NRC-CNRC), 1640a (National Institute of Standards and Technology), SPS-SW-1 (Spectrapure Standards), SPS-SW-2 (Spectrapure Standards) and TM-35 (Environment and Climate Change Canada) were used as certified reference materials. The acceptance of recovery was $\pm 10\%$ and all element recoveries were valid in this range. Germanium, rhodium and rhenium served as internal standards.

2.4.2. Identification of released organic compounds via NTS

The measurement of leachates was primarily performed using a non-target screening (NTS) approach (Nürenberg et al., 2015) with LC-QToF-MS consisting time of flight quadrupole mass spectrometry (TripleToF 6600, Sciex) coupled to a high-performance liquid chromatographic unit (1260 Infinity LC System, Agilent Technologies) with electrospray ionization (ESI). Ionization was carried in positive and negative mode. A reversed phase C18 column (ZORBAX RR Eclipse Plus C18, 2.1 mm x 150 mm, 3.5 μm , Agilent Technologies) equipped with a security guard (AQ C18, 2.0 mm x 4 mm, Phenomenex) was used as the stationary phase, and a binary mobile phase consisting of deionized water [A] and acetonitrile [B], each acidified with 0.1% formic acid. The gradient method has a total run time of 27 minutes. The flow rate was 0.3 ml min⁻¹ and column oven temperature was set to 40 °C. Three non-target specific internal standards Bezafibrate-d4 (CAS: 1189452-53-6, TRC, $\beta=1.0 \mu\text{g l}^{-1}$), Iopromide-d3 (CAS: 1189947-73-6, TRC, $\beta=2.0 \mu\text{g l}^{-1}$) and Olmesartan acid-d6 (CAS: 1185144-74-4, Biozol, $\beta=0.5 \mu\text{g l}^{-1}$) were added for quality assurance. The injection volume was 100 μl .

The NTS workflow included data files of the measurements which were analyzed with an NTS application based on R-script (Dietrich et al., 2022). The settings were adjusted with the following parameters for peak picking: mass range detection from m/z 100 – 1200 Da; m/z step: 0.02 Da; MS2 precursor m/z tolerance: 20 ppm; RT range from 2.2 min to 19.8 min. The peak width varied from 5 to 80.7 sec (minimum intensity: 5 cps; signal to noise ratio S/N: 3; noise (scans): 30 cps; maximum peaks per peak: 10). Alignment was performed to sum up only group leaders and using internal blank correction.

The identification of substances during NTS was based on the level system of Schymanski et al. (2014a), Schymanski et al. (2014b). Unknown features with relatively high intensities were an advice of Level 5: Exact mass of interest and a possible Level 4 classification: Unequivocal molecular formula by knowing the exact monoisotopic mass-to-charge ratio (m/z) and using e.g. enviPat Web (envipat.eawag.ch) to exclude possible isotopes and adducts. Experimental MS¹ and MS² data provided a first indication of Level 3: tentative candidate. Comparing MS¹ and MS² spectra from experimental data with compound databases e.g. Mass Bank (massbank.eu), m/z cloud (mzcloud.org), PubChem (pubchem.ncbi.nlm.nih.gov) and laboratory internal mass spectrum database (Jewell et al., 2020), it was possible to verify a possible compound and categorize it as Level 2: probable structure. With the help of a measured standard, the feature was set as Level 1: confirmed structure of a substance if matched with experimental data MS¹, MS² and retention time (RT) from NTS. Measurement of substance standards was performed with different collision energies (CE) 10-150 V in steps of 10 V in both ionization modes to achieve a range of fragment intensities.

2.4.3. Target method

Quantification was performed with LC-MS/MS consisting of Triple Quad (6500+ LC-System, Sciex) coupled with a high-performance liquid chromatographic unit (1260 Infinity LC System, Agilent Technologies) in the negative multiple reaction monitoring (MRM) mode. The column, chromatographic and mass spectrometric parameters were transferred from the non-target method. The gradient started with an isocratic step of one minute (A: 98%|B: 2%) and was followed by a linear gradient of (A: 80%|B: 20%) at 2 minutes, continuing with a slow increase of eluent B from 20% to 100% within 14.5 minutes. This ratio remains for 5.5 minutes and ends with fast step to the initial conditions (A: 98%|B: 02%) within 0.1 minutes and remained isocratic for 5 minutes. The injection volume was 10 μl . The 15-point calibration curve shows linearity for the target analyte and was created with range from 0.5 $\mu\text{g l}^{-1}$ to 160 $\mu\text{g l}^{-1}$ ($R^2 = 0.9998$; weighting: 1/x). Limit of quantification (LOQ) was set to 0.3 $\mu\text{g l}^{-1}$ (limit of detection (LOD): 0.08 $\mu\text{g l}^{-1}$) using validation data according to DIN

32645 (DIN, 2008) with linear 10-point calibration ($R^2=0.9999$; $n=3$; $k=3$; $CI=95\%$; procedural standard deviation $s_x=0.06$). The evaluation and quantification of target measurement was performed via device-specific software (MultiQuant 3.0.3, Sciex). Leachate triplicates were spiked with $10 \mu\text{g l}^{-1}$ standard solution of quantified target analyte for recovery experiments. Geotextile I [PET/PP] leachate L3 (2.25 d) recovery is $106 \pm 7.8\%$ and Geotextile II [PP] shows recovery of $124 \pm 17.6\%$.

2.4.4. Biotests

The leachates were tested for acute toxicity to bacteria, green algae and daphnids. In addition, mutagenic and estrogen-like effects were examined as described below.

Luminescent bacteria test: The procedure was adapted from ISO 11348-2 (DIN, 2023) and performed with freshly prepared bacteria (*Aliivibrio fischeri*) in white 96-well microtiter plates (μ CLEAR, Greiner Bio-One). The assay utilizes the bioluminescence and quantifies the inhibition of bacterial light emission after exposure to test sample as a measure for acute bacterial toxicity. Prior to testing, the aqueous leachates were salinated with NaCl solution (20%, m/v) to a salinity value of 20. Methanolic solutions of identified compounds were pre-diluted 1:50 and simultaneously salinated with NaCl solution (2%, m/v). Each sample was tested in dilution series in a geometric sequence, threefold independently with three technical replicates each. A NaCl solution (2%, m/v) was used as negative control and 3,5-dichlorophenol (97%, Sigma, $\beta = 4.5 \text{ mg l}^{-1}$) served as a positive control. The lowest ineffective dilution (LID) that causes an inhibition of luminescence of less than 20% was used as the test result (DiBt, 2011). The statistical analysis of bioassay data was performed using the open-source software R (version 3.4.3). The fits of concentration-response relationships and estimates of EC_{50} were generated by a five-parameter log-logistic function (1):

$$f(x) = c + \frac{d-c}{(1+\exp(b \times (\log(x) - \log(e))))^f} \quad (1)$$

The response of bioassays was evaluated as a function of concentration x with the parameters c and d as lower and upper response limits respectively. The parameter e is defined as the inflection point, parameter b denotes the relative slope and the parameter f describes the asymmetry of the curve. Luminescence bacteria assay was performed with leachates of DSLT and with non-target identified single substances.

Algal growth inhibition test: The inhibition of the green alga *Desmodesmus subspicatus* (SAG 86.81) was examined in accordance with DIN 38412-33 (DIN, 1991). The incubation was performed in test vessels with a testing volume of 4 ml in a temperature-controlled cabinet at $23 \text{ }^\circ\text{C}$ under continuous white light ($60\text{-}120 \mu\text{E m}^{-2} \text{ s}$). Each sample was tested in dilution series in a geometric sequence and with three technical replicates. The density of algae at the beginning of the test was adjusted to $1 \times 10^4 \text{ cells ml}^{-1}$. Distilled water was used as a negative control and potassium dichromate ($\geq 95\%$, Merck, 0.5 mg l^{-1}) served as a positive control. After 72 h, the chlorophyll fluorescence of test cultures was measured with a spectrophotometer (Hitachi F-2500) at 685 nm and was used to calculate growth inhibition relative to control cultures grown under identical conditions.

Daphnia immobilization test: The inhibition of the mobility of the crustacean *Daphnia magna* (clone B) was investigated according to DIN EN ISO 6341 (DIN, 2013). For this purpose, five juvenile test organisms each were placed in 20 ml test solution in loosely covered beakers and incubated at $20 \pm 2 \text{ }^\circ\text{C}$ in the dark. Each sample was tested in dilution series in a geometric sequence and with two technical replicates. A solution of $293.8 \text{ mg l}^{-1} \text{ CaCl}_2 \times 2 \text{ H}_2\text{O}$, $123.3 \text{ mg l}^{-1} \text{ MgSO}_4 \times 7 \text{ H}_2\text{O}$, $5.8 \text{ mg l}^{-1} \text{ KCl}$ and $64.8 \text{ mg l}^{-1} \text{ NaHCO}_3$ was used to prepare the dilution series and also served as a negative control. After 48 h, the number of immobilized daphnids was recorded in all test vessels.

Ames fluctuation test: The mutagenicity of DMSO extracts was determined with the *Salmonella typhimurium* strains YG 1041 (frameshift tester strain) and YG 1042 (base-pair substitution strain) with the Ames fluctuation test according to ISO 11350:2012 (ISO, 2012). The mutagenic potential is determined by the increase of mutants that reverted to a histidine-independent growth in the presence of the test sample. The test strains were incubated in the presence of ampicillin (sodium salt, Sigma-Aldrich) and kanamycin (solution from *Streptomyces kanamyceticus*, 50 mg ml⁻¹ in 0.9% NaCl, BioReagent, Sigma- Aldrich). The incubation time of the strain YG 1041 in 384 well plates (Greiner Bio-One) was prolonged from 48 h up to 72 h due to a low number of wells with revertant growth in the positive controls. All samples were tested with and without metabolic activation using 2-aminoanthracene (96%, Sigma-Aldrich; $c = 4.14 \times 10^8 \text{ mol l}^{-1}$) and 2-nitrofluorene (98%, Sigma-Aldrich; $c = 3.78 \times 10^8 \text{ mol l}^{-1}$) as a positive control respectively. Distilled water served as the negative control. The cell density of the overnight cultures was adjusted to 150 FAU (Formazine Attenuation Units) for YG 1041 and 160 FAU for YG 1042 with S9 mix (from rat liver, Harlan Cytotest Cell Research GmbH). Without S9 mix, 170 FAU for YG 1041 and 80 FAU for YG 1042 were applied. A test sample was regarded as mutagenic if a significant increase in the number of revertant wells was observed compared to the negative control in at least one strain with or without S9 mix.

Yeast estrogen screen (YES): The estrogenicity of ethanolic extracts was investigated according to ISO 19040-1 (ISO, 2018) using the test strain according to (McDonnell et al., 1991a; McDonnell et al., 1991b) that is based on the strain *Saccharomyces cerevisiae* BJ3505 (protease deficient, MAT α , PEP4:HIS3, prb-1- δ 1.6 R, HIS3- δ 200, lys2-801, trp1- δ 101, ura3-52gal2can1). To detect the estrogenic potential of leachates, the respective cells were exposed at 30 °C for 18 h to the samples, followed by an activation of the human estrogen receptor alpha (ER α), in case of the presence of corresponding agonists in the sample. The activation of the receptor is measured by a reporter gene assay using the *lacZ*-gene – encoding the enzyme β -galactosidase – as the reporting element. Each leachate was tested with four technical replicates in 96-well microtiter plates (Cellstar, Greiner Bio-One). Distilled water was used as a negative control. A dilution series of 17 β -estradiol (E2; $\geq 98\%$, Sigma-Aldrich; $\rho = 500 - 0.66 \text{ ng l}^{-1}$) served as a positive control and for calibration in the yeast estrogen screen.

3. Results and discussion

3.1. Polymer characterization

ATR-FTIR analysis of single polymer fibers confirmed that they are single-sort fibers of polyester and polyolefins (spectra are shown in SI Figure S1). It was confirmed that Geotextile I consists of nonwoven PET and PP fibers and Geotextile II consists entirely of PP filaments. These individual fibers were needle-punched to form the synthetic fleece. This confirms that virgin material is used as the starting material for the production of the fibers of the nonwoven products. Typical compositions of polyolefin PP and polyester PET-containing geotextiles are specified with 85-99 weight percentage (wt%) of polymer resin with additional filler (0-13 wt%), carbon black pigments (0-4 wt%) and additives such as stabilizers, antioxidants, colorants and processing chemicals (0.1-2 wt%) (Hsuan et al., 2008; Wiewel and Lamoree, 2016). PP and PET materials have different levels of environmental persistence. For instance, geotextile materials of PP are more resistant to acidic and alkalic pH-values than PET (Markiewicz et al., 2022).

3.2. Multi-elemental analysis

The elements Al, P, S, Ti, Cr, Fe, Ni, Cu and Zn were detected in both geotextile materials after an oxidative microwave digestion, while Mn and Sb were only detected in Geotextile I [PET/PP]. The concentrations of V, Co, As, Se, Mo, Ag, Cd, Sn, Tl, Pb and U were below the LOQs (see SI Table S1). Elevated concentrations of Al and Ti were found in Geotextile I [PET/PP]. However, it is speculation as to whether the PET content or the use of certain catalysts during manufacturing of different PP products is causing these results. It is known that Al and Ti are applied as catalysts during the manufacture of polypropylene (Antinucci et al., 2024; Kioka et al., 1994). Sb was exclusively detected in Geotextile I [PET/PP], which mainly consists of PET. Sb_2O_3 is used during polymerization of PET as the most common polycondensation catalyst (Keresztes et al., 2009; Schildroth et al., 2021) and thus should be well distributed in the polymer. In the case of commercially available PET resin the concentration of Sb is between 190 and 300 $\mu\text{g g}^{-1}$ (Duh, 2002) in contrast to detected concentration of 100 $\mu\text{g g}^{-1}$ in digestion of Geotextile I [PET/PP].

The detected element concentrations in the leachates of the DSLT confirmed the release of the elements present in the bulk material of the geotextiles. Detected elements in digestion were presented in Figure 1) and most of them were released in elevated concentrations within the first three leaching steps. Element levels were highest in L1 (0.25 d) and decreased fast in the subsequent leaching steps L2 (1 d) and L3 (2.25 d). The conductivity of the first leachate L1 (Geotextile I [PET/PP]: 10 $\mu\text{S cm}^{-1}$; Geotextile II [PP]: 27 $\mu\text{S cm}^{-1}$) correlates with the initial release of elements and decreases to L4 and therefore on the blank level (1 $\mu\text{S cm}^{-1}$). Release of the most elements is elevated in leachates of Geotextile I [PET/PP] compared to Geotextile II [PP] released higher concentrations for most elements except for S, Cr, Fe and Cu. This different release in leachates of both materials might be explained by different polymer compositions or polymers used from different polymer processing. The measured release of the elements in DSLT fractions was probably caused by an initial wash-off with subsequent exhaustive leaching. This may be caused by the fiber structure, which increases the surface area of the geotextiles in full contact with the leaching agent. Since the material is made of non-reactive polymers, there is probably no appreciable interaction or retention of the elements in the geotextile material (Lassabatere et al., 2004). This favors the initial release of the elements. Elevated release of Al, P, Ti and Sb in leachates of Geotextile I [PET/PP] than Geotextile II [PP] also correlates very well with the results of the digestion experiment.

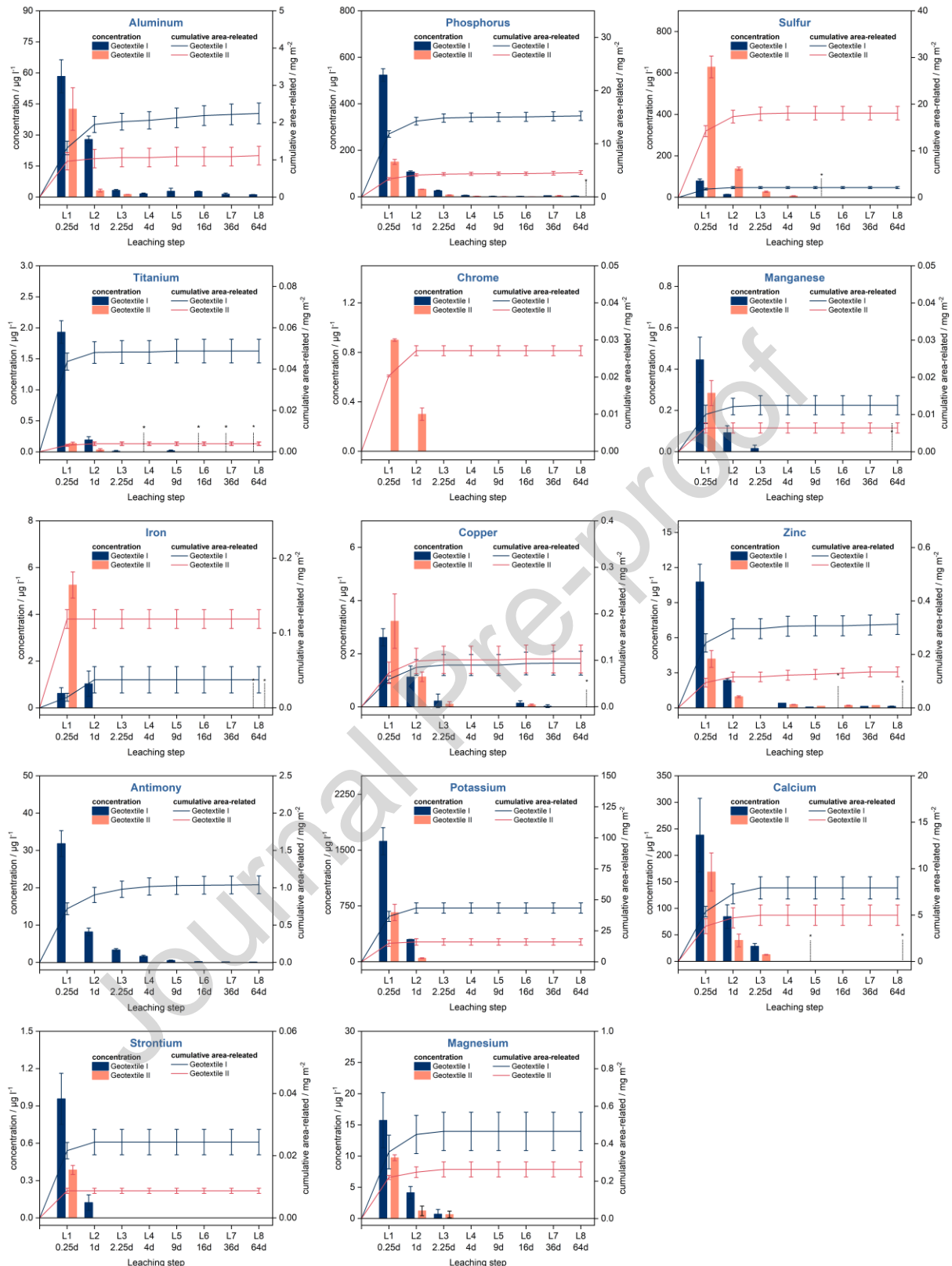


Figure 1. Elemental concentrations and cumulative area-related release from Geotextile I [PET/PP] and Geotextile II [PP] in leachates of DSLT L1 (0.25 d) - L8 (64 d), mean, $n=3$, error bars indicate standard error SE (\pm), values are blank corrected, * indicates outliers, LOQ: SI Table S2-1 to Table S2-3.

Exclusively Cr release is presented in Geotextile II [PP]. Levels of P, S, Zn in the blank control are clearly below the leachate levels. Additional findings of Na, Si, V, Ni, As, Mo, Sn, Cd and Pb were very probably increased by contaminations of vessels or filters of the deionized water device, since the

elements were also present in the blank samples with comparable concentration levels (see SI Chapter S3). Co, Se, Ag, Tl and U were not detected (<LOQ).

Elevated P levels indicate the presence of phosphate-containing compounds, for example the identified Tris(2,4-di-tert-butylphenyl) phosphite or monododecyl phosphate (MDP) (see chapter 3.3.2.) in leachates of Geotextile I [PET/PP] which showed three times stronger release of P, which is 24% of the total amount in Geotextile I [PET/PP] and 4% in Geotextile II [PP].

S concentration levels were more than seven times higher in leachates of Geotextile II [PP], indicating the presence of sulfur-containing organic compounds such as sulfonates, which were also found in NTS (see chapter 3.3.2.). S was also reported in residues of polypropylene from polymer catalysis (Hernandez-Fernandez et al., 2022).

Ca and K act as counter anions for surfactants and Sr as part of the technical calcium (Burger and Lichtscheidl, 2019; Pors Nielsen, 2004) possible in calcium stearate (Zhang et al., 2023). K is used as a process stabilizer and for thermal resistance in the form of potassium iodide in manufacturing in combination with copper salts and Mn could be complexed in UV stabilizer compounds. (Wiewel and Lamoree, 2016). $MgCl_2$ is also used as a supporting catalyst in plastic processing of PP (Bahri-Laleh et al., 2011; Hsuan, 2016), while Zn is used as zinc stearate $C_{36}H_{70}O_4Zn$ as a lubricant additive in plastic manufacturing and as an antistatic agent (Kulikov et al., 2009; Zhang et al., 2023). Calcium and zinc stearate were described as a processing stabilizer as part of PP containing geotextiles (Wiewel and Lamoree, 2016), but could not be detected in NTS.

Zn levels were analyzed in leachates of both geotextile materials with concentrations between $4 \mu g l^{-1}$ to $10 \mu g l^{-1}$ (with a total initial release of (0.1% - 0.5% of the total amount in the material)). So, values are below the level $14.4 \mu g l^{-1}$ of Predicted no effect concentration (PNEC) for freshwater (ECHA_a, 2024; Van Sprang et al., 2009). Considering further dilution processes under environmental conditions, the measured Zn concentrations should not have a significant effect in the environment. Nevertheless, Zn is highly bioavailable for water organism (El-Agawany and Kaamouh, 2023) and might cause toxic effects in them. Gartiser et al. (2017a) noted that the toxicity of Zn depends for example on pH or water-soluble form. They show Zn concentrations of up to $300 \mu g l^{-1}$ in leachates of their building products, which could possibly explain some toxicity in some of the tested species. Release of Sb was found exclusively in leachates of Geotextile I [PET/PP] and a decreasing trend with notable concentrations in the first leaching steps. Gartiser et al. (2017a) expected Sb release in their investigated PET multifil geotextile and PA/PP geocomposite. Westerhoff et al. (2008) observed Sb leaching from PET plastic water bottles and verified that only a small fraction of Sb releases from material into water. Comparison with the analysis of the fully digested material demonstrates that only 1.4% of the total amount in the material was released in the first leaching step. Due to the use of Sb_2O_3 as catalyst during PET polymerization, Sb is well distributed in PET and the assumption was on diffusion-controlled release. A PNEC value is provided by ECHA for Sb_2O_3 ($135 \mu g l^{-1}$) for fresh water organisms (ECHA_b, 2024). In view of further dilution in the environment the determined release of Sb should not lead to measurable environmental effects.

3.3. Release of organic substances and ecotoxicological effects

3.3.1. Dissolved Organic Carbon (DOC)

DOC was determined in leachates of tested geotextiles (basic chemical parameters are presented in SI Table. S4 and Table S5). The comparison of both geotextile leachates illustrates that the DOC release exhibited a similar trend (Figure 2). Geotextile II [PP] released initial $70.5 \pm 3.3 \text{ mg l}^{-1}\text{C}$, Geotextile I [PET/PP] $15.6 \pm 0.4 \text{ mg l}^{-1}\text{C}$ and DOC concentrations subsequently decreased in the first three steps below the LOQ, while Geotextile II [PP] concentrations are slightly above LOQ during full leaching period. The release mechanism of the geotextiles is probably caused by wash-off from the surface. With regard to the DOC results, it can be assumed that the largest amount of organic substances leaches out of the material in the beginning, which implicates that no continuous diffusion-controlled release occurs.

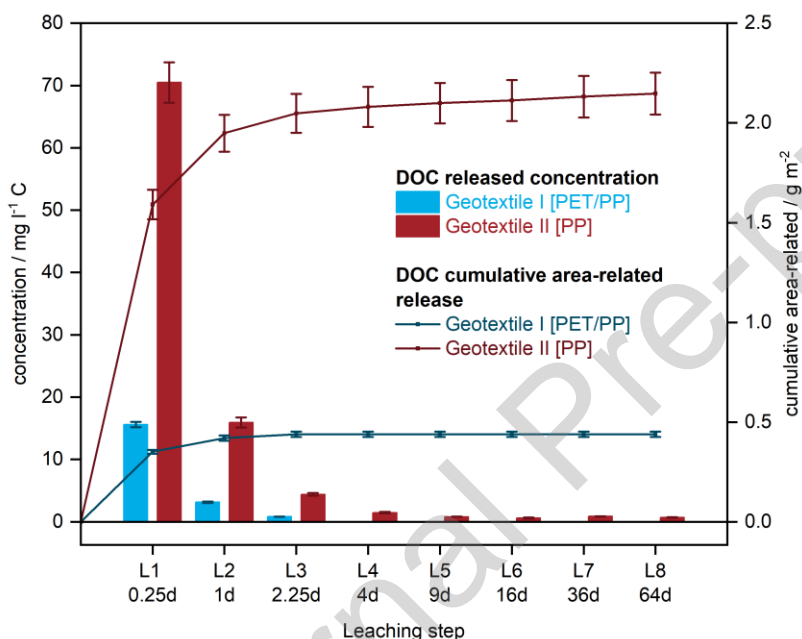


Figure 2. DOC level in leachates of Geotextile I [PET/PP] and Geotextile II [PP] showing total concentration of DOC in the leachates (LOQ=0.5 mg l⁻¹) and cumulative area-related release, DSLT L1 (0.25d) – L8 (64d), mean, n=3, error bars indicate standard error SE (±), values are blank corrected, LOQ: 0.5 mg l⁻¹.

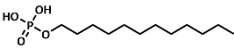
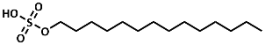
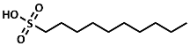
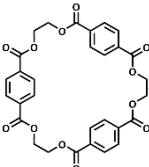
3.3.2. Non-target screening (NTS) of organic compounds


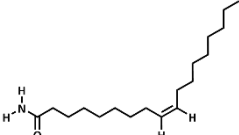
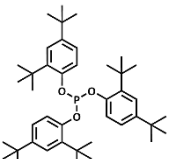

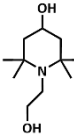
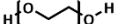
NTS was used to identify possible released organic compounds in leachates of geotextiles. Various organic compounds were detected in the leachates (Table. 1) and some of which occur exclusively in a certain geotextile material, possibly due to the different composition, origin of the processed polymer, needle-punched fibers and additives used.

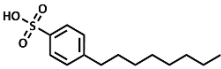
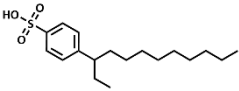
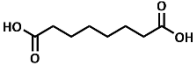
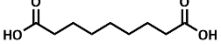
After peak picking, annotation via group leader and blank cleanup in the positive ionization mode for Geotextile I [PET/PP] and Geotextile II [PP] in L1 (0.25 d), initially 3,347 and 4,338 components were found, respectively. The numbers decreased in L8 (64 d) to 1,078 and 1,275 components, respectively. In the negative mode for Geotextile I [PET/PP] and Geotextile II [PP] in L1, initially 3,699 and 4,196 components were detected, respectively, which decline in L8 to 458 and 1,275 components, respectively (see SI Chapter S5 for information about components and intensities). The workflow of compound identification was carried out using spectral libraries and web databases. The identified substances were mainly surfactants. But antioxidants, light stabilizer, polyethylene glycols (PEG), series of PET oligomers, carboxylic acid amide and dicarboxylic acids were found, too. Reliably identified compounds are summarized and listed in Table 1 showing information about mass

spectrometry analysis, identification level, application and intensity trends. All substances presented were successfully categorized at Level 1 or Level 2. The intensities of all identified compounds decrease from first leaching step L1 (0.25 d) to individual leaching step where the compound signal transitions to signal to noise ratio. Polyethylene glycols were identified in the positive ionization mode [M+H], due to its typical repeated fragment m/z 44.03 Da (C₂H₄O). In total, twelve PEGs were found from triethylene glycol (PEG-3) m/z 239.1494 Da to PEG-15 m/z 679.4116 Da. The presence of PEGs repeat unit <3 in the leachates can also be assumed. Molecules around m/z 100 Da such as diethylene glycol (PEG-2) and ethylene glycol cannot clearly be detected using the NTS LC-QToF-MS method because these molecules are too small in size. With respect to the retention time, they appear in the dead time. Some additives were found such as antioxidant Tris(2,4-di-tert-butylphenyl) phosphite (Irgafos 168) and HALS 4-Hydroxy-1-(2-hydroxyethyl)-2,2,6,6-tetramethylpiperidine (HA 201). Irgafos 168 is described as an additive of PP geotextiles causing no toxic effects to aquatic organism, does not cause bioaccumulation, but is not readily biodegradable, while several HALS compound are described with proportion of identified piperidone compounds (Wiewel and Lamoree, 2016). PEGs were detected in leachates of both geotextile materials but the intensity is higher in Geotextile II [PP]. PET oligomers were only detected in leachates of PET-containing Geotextile I [PET/PP] in positive ionization mode [M+H], especially series of cyclic trimers and dimers, e.g. identification of ethylene terephthalate cyclic trimer. In negative ionization mode [M-H], surfactants containing benzenesulfonates such as dodecylbenzene sulfonate, sulfates such as tetradecyl sulfate and phosphates like dodecyl phosphate were identified as antistatic lubricant additive with different long aliphatic chains. Lubricant slip additive oleamide was found in leachates of both products in large quantities, as well as additives suberic and azelaic acid. Lubricants prevent adhesion to processing equipment, improve fluidity and they reduce surface friction (Bridson et al., 2021). Phosphate ester monododecyl phosphate (MDP) were identified in leachates of Geotextile I [PET/PP] with levied intensities.

Table 1. Identified organic compounds via non-target screening (NTS) workflow in DSLT of leachates L1 (0.25 d) - L8 (64 d) from Geotextile I [PET/PP] and Geotextile II [PP]. The table indicates: compound information and structure, neutral monoisotopic mass, precursor ion and the prioritized polarity (MS¹), fragments in MRM daughter ion (MS²), retention time (RT), category via Identification level (LEVEL 5 – LEVEL 1), detection of the compound in geotextile material; intensity and trend: ↓ indicates that intensity decreases continuously over leaching time; ↑ indicates that intensity increases continuously over leaching time, n=3, mean, intensities are blank corrected.

Compound information and application	Compound structure	Monoisotopic mass Da	MS ¹ (ESI) m/z Da	MS ² m/z Da	RT min	identification level (Schymanski et al., 2014a)	detected in Geotextile I or/and Geotextile II leaching step detection (intensity and trend) ↓ ↑ cps
Monododecyl phosphate C12H27O4P lubricant additive		266.1647	[M-H] 265.157 4	78.9595 62.9649 96.9701	13. 9	LEVEL 1	Geotextile I [PET/PP] L1 (17,540,478) - L6 (383,539) ↓
Tetradecyl sulfate C14H30O4S antistatic agent		294.1865	[M-H] 293.179 2	97.2873 79.9145 95.8451	15. 3	LEVEL 1	Geotextile II [PP] L1 (29,017,054) - L4 (4,335,556) ↓
Decane sulfonate C10H22O3S antistatic agent		222.1290	[M-H] 221.121 7	79.9571 80.9646 63.9616	11. 5	LEVEL 1	Geotextile II [PP] L1 (16,289) - L3 (666) ↓
Ethylene terephthalate cyclic trimer C30H24O12 PET oligomer		576.1268	[M+H] 577.134 1	149.031 8 193.054 0 385.082 7	14. 5	LEVEL 1	Geotextile I [PET/PP] L1 (1,214,395) - L7 (1,620) ↓

Ethylene terephthalate cyclic dimer C20H16O8 PET oligomer		384.0845	[M+H] 385.0918	195.0943 179.0994 123.0386	9.7	LEVEL 1	Geotextile I [PET/PP] L1 (182,849) - L8 (1,096) ↓	
Oleamide C18H35NO lubricant slip additive (primary)		281.2719	[M+H] 282.2794	69.0701 97.1009 83.0928	18. 5	LEVEL 1	Geotextile I [PET/PP] L1 (5,838,721) - L7 (19,161) ↓	Geotextile II [PP] L1 (8,616,250) - L5 (12,310) ↓
Tris(2,4-di-tert-butylphenyl) phosphite C42H63O3P Antioxidant stabilizer (secondary) Irgafos 168		646.4515	[M+H] 647.4588	147.5931 235.6328 291.4821	17. 4	LEVEL 1	Geotextile I [PET/PP] L1 (3,787,849) - L4 (67,022) ↓	Geotextile II [PP] L1 (144,607) - L5 (2,786) ↓
2,2,6,6-Tetramethyl-4-piperidone C9H17NO Polymer stabilizer (HAS)		155.1310	[M+H] 156.1381	58.0746 42.0340 39.0228	2.8	LEVEL 2	Geotextile II [PP] L1 (20,436) - L8 (13,167) ↓	
4-Hydroxy-1-(2-hydroxyethyl)-2,2,6,6-tetramethylpiperidine C11H23NO2 Light stabilizer (HALS) HA-201		201.1728	[M+H] 202.1802	102.0994 84.0890 44.0582	2.4	LEVEL 2	Geotextile II [PP] L1 (767,358) - L8 (95,770) ↓	
Poly ethylene glycol polymers (PEGs) (C2H5O) _n OH PET process chemical, lubricant								
PEG-3	C6H14O4	150.0892	[M+H] 151.096	45.0464	4.2	LEVEL 1	Geotextile I [PET/PP] L1 (1,038) -	Geotextile II [PP] L1 (3,722) -
PEG-4	C8H18O5	194.1154	5	89.0650 [from	4.8		L4 (239) ↓	L3 (581) ↓
PEG-5	C10H22O6	238.1416	195.122	PEG-4]	5.0	[PEG-3]	L1 (5,6232)	L1 (284,069)
PEG-6	C12H26O7	282.1679	7	133.0968 [from	5.2	to	- L4	- L8
PEG-7	C14H30O8	326.1941	239.148	PEG-7]	5.4	[PEG-8]	(19,250) ↓	(43,676) ↓
PEG-8	C16H34O9	370.2203	9	177.1171 [from	5.5		L1 (710,843)	L1
PEG-9	C18H38O10	414.2465	283.175	PEG-8]	5.7	LEVEL 2	- L8	(10,785,580
PEG-10	C20H42O11	458.2727	1		5.8		(317,994) ↓) - L8
PEG-11	C22H46O12	502.2989	327.201		6.0	[PEG-9]	L1	(747,995) ↓
PEG-12	C24H50O13	546.3251	3		6.1	to	(6,377,127)	L1
PEG-13	C26H54O14	590.3514	371.227		6.2	[PEG-15]	- L8	(41,724,576
PEG-14	C28H58O15	634.3776	6		6.4		(498,012) ↓) - L8
PEG-15	C30H62O16	678.4038	415.253		6.5		L1	(1,904,104)
			8				(14,089,307	↓
			0) - L8	↓
			503.306				(530,190) ↓	(61,287,529
			2				L1) - L8
			547.332				(19,517,130	(1,611,384)
			4) - L8	↓
			591.357				(478,224) ↓	L1
			3				L1	(68,419,338
			635.384				(22,267,977) - L8
			8) - L8	(1,076,361)
			679.411				(432,350) ↓	↓
			1				L1	↓
							(21,158,945	(68,785,834
) - L8) - L8
							(413,291) ↓	(535,474) ↓
							L1	L1
							(19,359,317	(70,018,272
) - L8) - L8
							(309,652) ↓	(289,130) ↓
							L1	L1
							(14,648,591	(61,275,744
) - L8) - L8
							(242,106) ↓	(170,007) ↓

							L1 (4,568,972) – L7 (1,666) ↓ L1 (4,882,383) – L8 (1,051) ↓ L1 (2,399,448) – L6 (353) ↓	L1 (41,751,396) – L7 (82,990) ↓ L1 (22,294,538) – L4 (234,898) ↓ L1 (10,041,973) – L3 (572,720) ↓ L1 (4,611,080) – L8 (71,506) ↓
Octyl benzenesulfonate C14H22O3S antistatic agent		270.1290	[M-H] 269.1217	170.0059 183.0128 79.9576	14. 4	LEVEL 1	Geotextile I [PET/PP] L1 (67,914) – L5 (474) ↓	
Dodecyl benzenesulfonate C18H30O3S antistatic agent		326.1916	[M-H] 325.1436	119.9543 183.6832 80.1258	10. 5	LEVEL 1	Geotextile I [PET/PP] L1 (20,438) – L3 (787) ↓	
Suberic acid C8H14O4 lubricant additive		174.0892	[M-H] 173.0815	111.0816 83.0503 53.0505	6.5	LEVEL 2	Geotextile II [PP] L1 (908,256) – L8 (8,714) ↓	
Azelaic acid C9H16O4 lubricant additive		188.1049	[M-H] 187.0970	125.0984 97.0659 57.0348	7.2	LEVEL 2	Geotextile II [PP] L1 (5,397,252) – L8 (3,247) ↓	

3.3.3. Selection of bioassay

For screening of sensitive biological endpoints, samples of a preliminary static surface leaching test were investigated for the acute toxicity to bacteria, green algae and daphnids as well as estrogen-like and mutagenic effects (results are presented in Figure 3). Significant effects were only detected by the bioluminescence inhibition assay with *Aliivibrio fischeri*. Leachates of Geotextile I [PET/PP] caused a LID of 64, while Geotextile II [PP] shows a LID of 8. According to these results, only the luminescent bacteria assay was selected for biotesting of DSLT leachates. The luminescent bacteria assay is a standard test for the assessment of the release of dangerous substances from construction products and is proven for the sensitive detection of toxic substances (Heisterkamp et al., 2021).

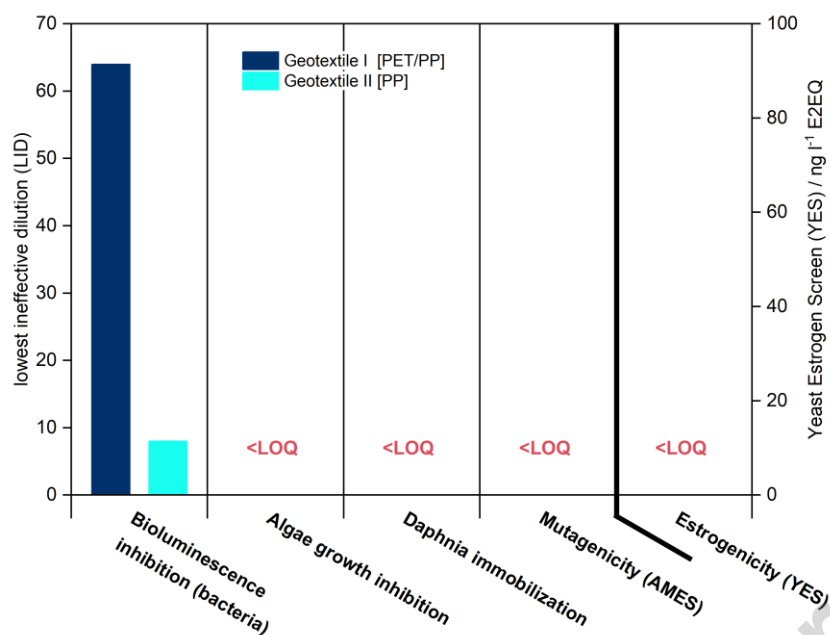


Figure 3. Toxicity to bacteria, algae and daphnids, mutagenicity and estrogenicity of leachates of Geotextile I [PET/PP] and Geotextile II [PP] in 28 days static surface leaching. LOQ: Algae test: LID = 1.2, Daphnia test: LID = 1, Bacteria: LID = 2.2, Ames: LID = 1.2, YES: 13.9 ng l⁻¹ E2EQ.

Leachates of Geotextile II [PP] showed no toxic effect in any biotest, although DOC release is five times higher than Geotextile I [PET/PP], while the latter exhibited an inhibition of bacteria *Aliivibrio fischeri*. By contrast, Gartiser et al. (2017a) investigated two geosynthetic materials and detected in leachates of PET multifil with polymer covering geotextile slightly evidence of toxicity in organism *Desmodesmus subspicatus* (LID = 4), *Daphnia magna* (LID = 2) and for *Aliivibrio fischeri* no effect. However, the other product, a PA monofilament/PP fleece coating material, shows toxicity in daphnia LID = 4 and bacteria LID = 8. is similar to the results of Geotextile II [PP] in Figure 3. Also comparable are results from Scholz et al. (2021) demonstrating that leachates of aged PP geotextile material shows no toxic effect to tested water organism *Desmodesmus subspicatus* and *Daphnia magna*.

3.3.4. Target analysis of MDP linked to bacteria bioassay

Substances identified in leachates of Geotextile I [PET/PP] show similar trend pattern of decreasing intensity compared to decreasing toxic effect during leaching period of DSLT like mono dodecyl phosphate (MDP), octyl benzenesulfonate, dodecyl benzenesulfonate, Oleamide, Irgafos 168, PET-oligomers and PEGs. Therefore, single substance tests with luminescent bacteria were carried out for these substances. With the exception of MDP, no substances tested exhibited any inhibition effect on luminescent bacteria.

MDP was identified by NTS at Level 1 and showed elevated intensities in leachates of Geotextile I [PET/PP] (see chapter 3.3.2.), which might indicate a strong occurrence of this substance in leachates. Based on these findings, target analytic of MDP was performed with both geotextile leachates by using transferred NTS-method on LC-MS/MS system. The substance was quantified only in leachates of Geotextile I [PET/PP] (see Figure 4A), while it was not detected in leachates of Geotextile II [PP] and not in blank group. Initially $4502 \pm 65 \mu\text{g l}^{-1}$ were detected in L1 (0.25 d). Subsequently the concentrations decreased until L6 (16 d). In L7 (36 d) and L8 (64 d) MDP was not detected at all. So, MDP also indicates wash-off from the material surface. Its occurrence is probably caused by its function as a lubricant in the manufacturing process to prevent adhesion to the needle

punching devices of the geotextile but it can also be applied in the plastic processing (Bérubé and Saunier, 2016; Bridson et al., 2021; Richter, 2009). The use of lubricants and MDP as avivage is also described in patent specification [see CN106868718B (2017); (WO1993002247A1, 1993)].

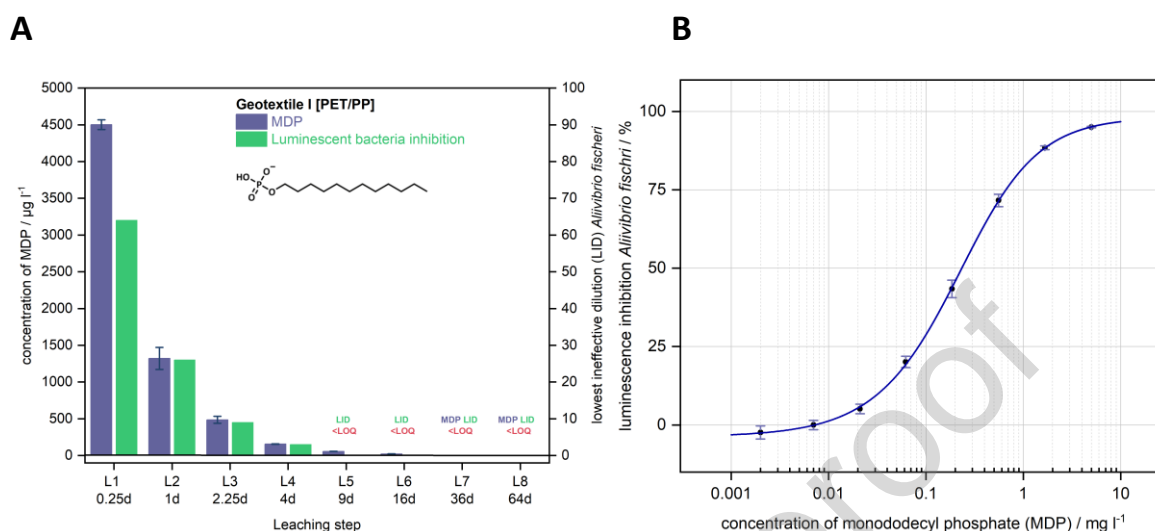


Figure 4. A Concentration of monododecyl phosphate (MDP) in leachates of Geotextile I [PET/PP] (mean, $n=3$, error bars indicate standard error SE (\pm), values are blank corrected, LOQ: $0.3 \mu\text{g l}^{-1}$) and toxic effect to *Aliivibrio fischeri* (LOQ: LID 2.2), DSLT L1 (0.25 d) - L8 (64 d) B represents the dose-response curve of single substance MDP in luminescent bacteria assay.

Inhibitory effects were identified in leachates of Geotextile I [PET/PP] with *Aliivibrio fischeri* (see Figure 4A), while no effects were found in leachates of Geotextile II [PP] (see SI Table S7). The results are combined with results of MDP analysis. Effects [LID = 64] in L1 (0.25 d) is in good accordance with results obtained from the 28-day static surface leaching test (Figure 3), which indicates a release of toxic substance in the first step. Decreasing bacteriotoxic trend correlates with the wash-off trend of MDP.

In order to calculate the percentage of the toxic effect in leachates, dose-response curves were carried out with MDP and with further single representative substances identified in NTS at Level 1 in the leachates of Geotextile I [PET/PP] (Table 1). Dose-response curves of organic substances with no effect are not presented. The dose-response curve of MDP, shown in Figure 4B, resulted in an EC_{50} of $210 \pm 7.5 \mu\text{g l}^{-1}$. Thus the measured MDP concentrations can cause an average of about 85% of the effect found in the bacterial assay. It is known from the literature that the similar compound dodecyl sulfate initiated the lysis on cell membranes (Grigorov et al., 2021) and bacteria cells (Asio et al., 2023; Woldringh and Van Iterson, 1972). It is likely that MDP has similar effects. In leachates of both the geotextiles, dodecyl sulfate was not found by NTS. The phosphorus concentration of $530 \pm 27 \mu\text{g l}^{-1}$ quantified by ICP-MS in L1 of Geotextile I [PET/PP] can be explained mainly by MDP which releases roughly $90.5 \pm 1.5\%$ of phosphorus as a proportion of the compound, while the cumulated carbon content of substance MDP accounts for 18% of the total DOC quantified.

3.4. Release in groundwater and river water

Leaching in deionized water is worst-case scenario for substance release. In order to investigate the impact and the transferability to environmental water matrices, groundwater (GW) and river water (RW) were used to study the different release behaviour in DSLT steps L1 (0.25 d) - L4 (4 d). When using deionized water, bacteriotoxicity was only detected in leachates of Geotextile I [PET/PP], which was therefore selected for these additional studies. DOC is shown in Figure 5, bacteria test and MDP analysis in Figure 6 and basic chemical parameters are presented in SI Table S10-3.

In GW and RW the release trends of elements are on the same scale as the release in deionized water (see SI Figure S5 and SI Table S10-4a to S10-4c). With regard to increased background concentrations in environmental waters, leachate concentrations and a release trend for the most elements is not recognizable (Na, Mg, Si, S, K, Ca, V, Cr, Mn, Ni, Cu, Zn, As, Se, Mo, Ag, Cd, Pb and U concentrations are on the blank level). Al and Sb leachate concentrations were found significantly higher than the background, but lower than in leachates using deionized water, which is attributed to a reduced concentration gradient of the environmental waters. A slightly increased release in GW and RW was observed for P and Fe for reasons not further investigated. All four elements show comparable releases in GW and RW.

Figure 5 illustrates the trend of DOC which shows up to 26% lower release in GW and 32% in RW than in deionized water ($15.6 \pm 0.4 \text{ mg l}^{-1} \text{ C}$) in the first step. The release in GW and RW converges and is slightly higher in GW. DOC decreases with similar concentrations in both waters during leaching period, but DOC is still present in GW L4.

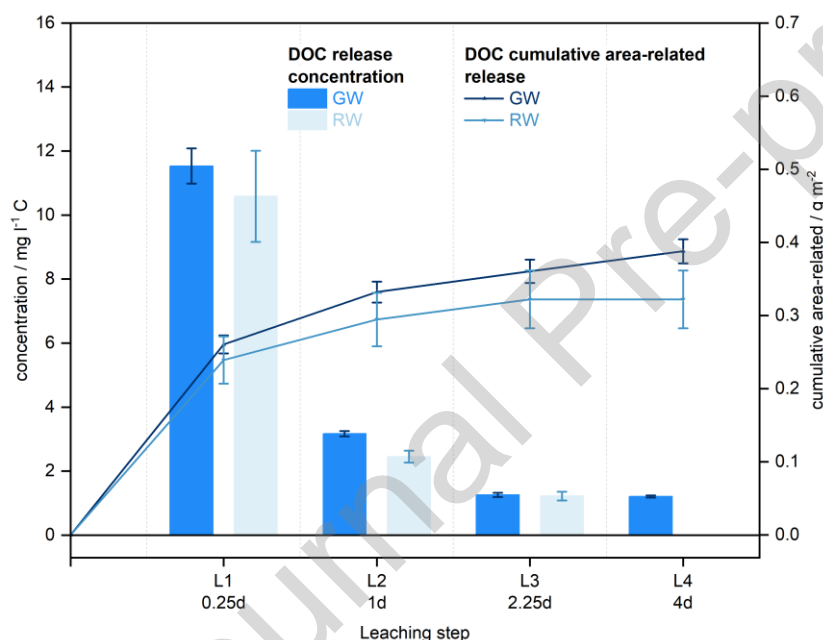


Figure 5. DOC level in leachates of Geotextile I [PET/PP] in groundwater (GW) and river water (RW). Total concentration and cumulative area-related release is presented, DSLT L1 (0.25 d) – L4 (4 d), mean, $n=3$, error bars indicate standard error SE (\pm), values are blank corrected, LOQ: 0.5 mg l^{-1} .

Figure 6 shows the correlation of a decreasing trend of MDP concentration in leachates of Geotextile I [PET/PP] combined with the decrease of bioluminescence inhibition of *Aliivibrio fischeri* in context of leaching with GW and RW. The initial release of MDP in the first leaching step is reduced in environmental water, by about 76% in GW (1.1 mg l^{-1}) and 84% (0.7 mg l^{-1}) in RW compared to deionized water (4.5 mg l^{-1}). This ratio continues in the further steps. Obviously, deionized water offers a stronger gradient for release of organic substances.

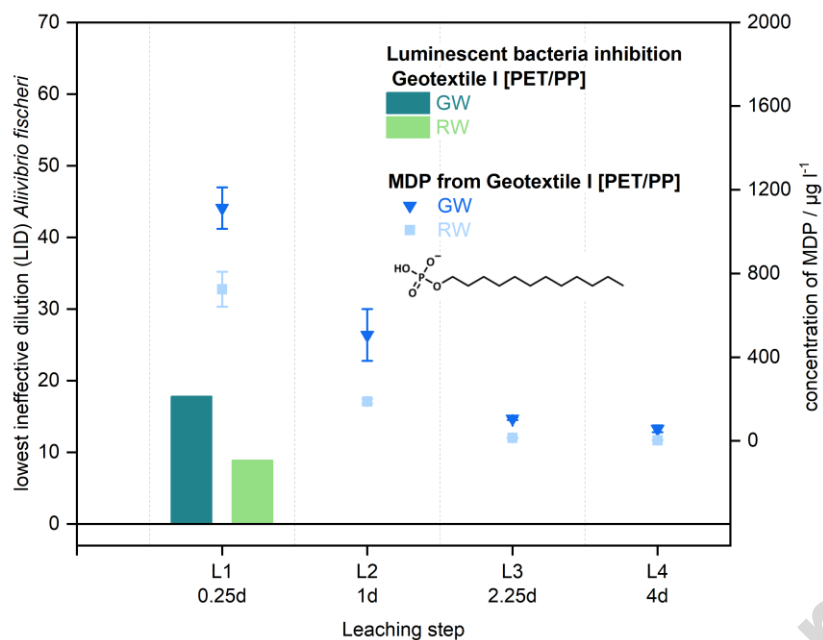


Figure 6. Toxicity of *Aliivibrio fischeri* in DSLT L1 (0.25 d) – L4 (4 d) of Geotextile I [PET/PP] in leachates of groundwater (GW) and river water (RW), effects at lowest ineffective dilution (LID), LOQ LID: 2.2, compared to released concentrations of MDP, values are blank corrected, mean, n=3, error bars indicate standard error SE (\pm), LOQ=0.5 $\mu\text{g l}^{-1}$.

Bacteriotoxic effect shows a similar but also reduced trend in environmental waters compared to deionized water (LID = 64). The toxic effect is not detectable anymore in GW and RW directly after the first leaching step.

Because of high background contaminations identification with NTS, determination of elements, DOC and MDP is preferable in deionized water. It is favorable matrix to investigate the release trend, release mechanism and for optimal non-target screening. However, it is useful for the assessment of environmental relevance the testing with groundwater and river water. The leaching experiments with GW and RW have demonstrated, that the results from the DSLT with deionized water are comparable and transferable to other matrices and to environmental scale. Compared to leaching with deionized water, the release of MDP to environmental waters and their bacteriotoxicity indicates a more reduced hazardous impact on the environment.

3.5. Environmental relevance

It was shown that MDP can be leached from geotextiles, which shows a toxic effect to the tested marine bacterium. Information about ecotoxicity of MDP is limited, but according to a REACH dossier the substance may cause long-lasting harmful effects to aquatic life (ECHA_c, 2024). And although observed released concentrations in environmental waters have been reduced compared to experiments in deionized water, MDP concentrations still represent 3% - 18% of the overall released DOC. Based on our findings it can be assumed, that the release mechanism is dominated by an initial wash-off mechanism, while diffusion-controlled release was not observed, thus suggesting potential short-term hazardous effects to the environment.

Luminescence inhibition tests with *Aliivibrio fischeri* demonstrated that 85% of the observed effects are caused by MDP. Potential effects caused by released heavy metals are regarded to be less important, as concentrations in leachates have been determined to be in the range of natural background concentrations in most cases. Furthermore, substances released from geotextiles in surface waters might experience larger dilution and a more frequent exchange of water compared to the laboratory study presented here.

Washing the geotextile material before installation would be a possible procedure to eliminate possible contaminants. This is also recommended by (Lassabatere et al., 2004). However, this means additional effort before material emplacement and possibly more technical equipment. So, the procedure can increase the cost and the wash water must also be collected as waste water.

4. Conclusion

The anionic surfactant monododecyl phosphate (MDP) was identified as an acute bacteriotoxic agent. The compound is used in elevated quantities as a lubricant in avivages during the manufacturing process and is obviously washed off from the material surface. In this context, DOC and most of the analyzed elements showed similar trends with a strong initial release and thus very probably wash-off from the surface. This behavior was observed in deionized water, but it was also verified reduced in environmental waters. In order to avoid the release of MDP into the aquatic environment, it should be replaced in manufacturing process with lubricants which do not exhibit ecotoxicological effects. An alternative procedure would be to wash the geotextile membranes prior to use. The results of this study confirm that investigation of the release of substances in combination with ecotoxicological biotests is recommended so as to identify environmental-friendly products. Furthermore, it can be concluded that the additives are more relevant than the basic polymer with regard to ecotoxicological effects. Currently, however, the release and formation of microplastic or nanoplastic fibers by physical stressors such as waves or the dismantling or replacement of geotextiles is unknown. Furthermore, it might be helpful to identify further compounds that are released by geotextiles and are responsible for the DOC measured.

Declaration of Competing Interest

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

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Appendix A. Supplementary data

Supplementary material data associated with this article can be found, in the online version, at doi:

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Declaration of interests

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

HIGHLIGHTS

- Leaching of contaminants from geotextiles is controlled by surface wash-off
- Early leachates of dynamic surface leaching test can cause high bacterial toxicity
- Identification of released organic compounds via non-target-screening
- Linking of chemical analysis with biotests
- Avivage agent monododecyl phosphate was identified as main driver of toxic effects