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Bisphenol A leaching from sewer renovation products

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ABSTRACT

This paper presents findings on the leaching behavior of bisphenol A (BPA) and its potential substitute, bisphenol F (BPF), from sewer renovation products. Deionized water was used as the leaching medium in experiments conducted using the Dynamic Surface Leaching Test (DSLT), in accordance with EN 16637-2:2023. Four resin formulations were evaluated, comprising two epoxy and two vinyl ester resin systems. BPA, a monomer, has been widely employed in sewer renovation products due to its excellent polymerization properties. For instance, in the United Kingdom, epoxy-based resin systems have fully replaced cement-based materials, such as mortar, for the rehabilitation of defective pipes since the late 1990s. However, concerns have emerged regarding the endocrine-disrupting properties of BPA, its potential impact on drinking water quality, and its broader environmental implications. Achieving the dual objectives of effective remediation and minimal release of hazardous substances presents a significant challenge. Collaboration among regulatory bodies, manufacturers, and researchers is essential to establish guidelines that balance material performance with environmental and health safety. This necessitates comprehensive testing and evaluation of materials to assess their long-term behavior and leaching potential. In this study, only one resin system was found to exceed the upper limit of 0.085 milligrams per square meter for cumulative BPA release in the DSLT, as determined by a predictive model and aligned with the Environmental Quality Standard threshold. Further investigations using ERα-CALUX and semi-quantitative gas chromatography/mass spectrometry (GC/MS) screening revealed that BPA is not the sole contributor to the toxicological effects observed in the eluate.

1. Introduction

1.1. The present status of the sewer system in European countries

In the study by Salihu et al. (Salihu et al., 2022), a multitude of factors contributing to the deterioration of materials used in aging pipes were examined, including a country's geographical characteristics and level of technological advancement. It is well established that metropolitan areas worldwide face substantial challenges due to the degradation of critical infrastructure (Macchiaroli et al., 2023, Dolores et al., 2022, Macchiaroli et al., 2023, Liu et al., 2023, Fasolino et al., 2016). The renovation of deteriorating underground sewer systems is of paramount importance, as it plays a vital role in improving urban environments and supporting the sustainable development of cities (Macchiaroli et al., 2023, Li et al., 2023). Sewer pipe failures pose significant environmental risks, potentially compromising the quality of air, water, and soil (Iurchenko et al., 2016). The leakage of substances from damaged pipes into surrounding soil and groundwater contributes to environmental pollution and negatively affects ecosystems. Typically, pipe rehabilitation is required after 35–45 years of service. A substantial portion of Germany's building stock was constructed during the post-World War II period, resulting in widespread infrastructure deterioration, a phenomenon common across many European countries. For example, in Sweden, it is estimated that approximately 471,000 apartments require renovation, including pipe repairs (Berglund et al., 2018). As a result, the demand for sewer renovation is increasing, with resin-based rehabilitation products emerging as a prominent solution. While concrete and stoneware remain the most commonly used materials in public sewer networks, the proportion of plastic pipes continues to grow (Berger et al., 2020). Germany has an extensive public sewerage network, spanning 594,334 kilometers, to which 97 % of the population is connected (RSV (Rohrleitungssanierungsverband) 2021). Inliner applications, where a new liner is inserted into existing pipes, have been developed to restore structural integrity in aging sewer systems.

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Trenchless technologies, such as Cured-In-Place Piping (CIPP) and sliplining, have gained popularity due to their ability to rehabilitate pipes without the need for extensive excavation. These methods have proven effective in sewer system remediation. However, a review of current research reveals a lack of scientifically robust studies on the short-term release of bisphenol A (BPA) following the installation of these resin-based products. Moreover, the long-term behavior and aging processes of epoxy linings in situ remain insufficiently understood (Rajasärkkä et al., 2016, Cantoni et al., 2021).

1.2. Resins for sewer renovation systems

Epoxy and vinyl ester resins have been identified as suitable materials for trenchless pipeline renovation (Wood, 1979, Hoffmann and Warren, 1999, Ellison et al., 2010, Pekkinen, 2011, Stratview Research Inc. 2017). The production of these resins typically involves the use of bisphenol A (BPA) to create the prepolymer, and in certain products, BPA is also used in the hardener component. BPA is a synthetic compound consisting of two equivalents of phenol and one equivalent of acetone, which accounts for the "A" in its name. Other bisphenols, such as bisphenol F, can also be used in resin formulations. The two hydroxyl groups of BPA enable the attachment of epichlorohydrin molecules to both ends of the bisphenol structure. Following dehydrohalogenation, bisphenol A diglycidyl ether, commonly referred to as a bis-epoxide, is produced. This bis-epoxide functions as a monomer (Component A) and is subsequently combined with a hardener (Component B), which typically consists of polyamines or phenolic compounds such as BPA itself (Pham and Marks, 2005). The hardener is added in a predetermined mixing ratio. If BPA is not fully incorporated into the resin matrix, there is potential for its release during use. In contrast, a well-formulated resin system exhibits minimal BPA release, whereas insufficient polymerization can result in elevated leaching rates, thereby posing a significant health hazard [(UBA (Umweltbundesamt) 2010, Amiridou and Voutsa, 2011, Husøy et al., 2019, Wang et al., 2022)].

The production of vinyl ester resins involves a prepolymer containing two or more acrylate or methacrylate groups, commonly referred to as vinyl groups. This prepolymer is typically synthesized from the previously mentioned bis-epoxide and acrylic acid. It is then dissolved in a suitable solvent, most commonly styrene. The curing process is initiated by the addition of a peroxide compound, or by the application of heat or UV light. In the context of sewer renovation, the use of UV light curing systems has been documented. Subsequent polymerization leads to the formation of a thermoset plastic. Although substantial quantities of BPA are used in the synthesis of epoxy and vinyl ester resins, it is generally assumed that the compound exists in its polymerized form and is therefore not readily released. However, the presence of residual monomer content cannot be entirely ruled out in practice.

1.3. Concerns related to BPA and other bisphenols in the context of sewer renovation

The issue of BPA leaching from materials used in sewer rehabilitation products, including liner systems, has gained increasing attention, particularly since BPA was added to the REACH Candidate List in January 2018. This inclusion underscores concerns regarding BPA's harmful effects on human health and its environmental impact [(UBA (Umweltbundesamt) 2010, ChemSec 2011)]. While previous research on BPA leaching from sewer rehabilitation products has been informative, it remains incomplete. For example, the International Chemical Secretariat (ChemSec 2011) reported BPA concentrations of up to 30 μg/L in water when liner systems were exposed to temperatures above 70 °C. Similarly, Rajasärkkä et al. (Rajasärkkä et al., 2016) observed elevated BPA concentrations in eluates with increasing temperature. However, most studies have not investigated the first week following installation, a critical period for understanding initial leaching dynamics (Rajasärkkä et al., 2016, Cantoni et al., 2021, Bae et al., 2022, Bruchet et al., 2014, Kosaka et al., 2012). Furthermore, there is a notable lack of studies specifically addressing the leaching behavior of BPA in real drinking water distribution networks (DWDNs), despite its identification as a potential contaminant in such systems due to the use of liner technologies. Comprehensive research is needed to advance understanding of the leaching mechanisms of BPA under a wider range of environmental conditions, particularly in soil and water systems, and to assess its short- and long-term impacts on water quality and human health.

1.4. Experimental approach to investigate the environmental impact of liners

A variety of test methods have been developed to assess the leaching behavior of building materials. Many of these methods are designed specifically for granular materials, such as batch tests according to the EN 12457 standard series (EN 12457-4 2022) or percolation tests, e.g., EN 16637-3 (EN 16637-3 2023). However, these tests are not suitable for liners and grouts, as they require the material to be shredded, an action that introduces new surfaces and leads to unrealistic leaching concentrations. As a result, two alternative approaches are available for testing resins used in liners or grouts. The first involves a modified percolation test, in which the resin is injected into a column filled with sand, and water is allowed to flow around the injected material (DIN 19631 2016). The second approach is a tank leaching test, where the solid material is immersed in a liquid without flow, and the liquid is replaced at predefined time intervals. It is important to note that these two approaches produce differing results when evaluating leaching behavior. Brameshuber and Vollpracht (Brameshuber and Vollpracht, 2011) compared the outcomes of the injection percolation test, as specified in DIN 19631 (DIN 19631 2016), with those of the Dynamic Surface Leaching Test (DSLT), a tank test defined in EN 16637-2:2023 (EN 16637-2 2023). Their findings indicate that the flow rate and/or the movement of water around the material in the injection percolation test significantly affects the leaching of substances from the resins compared to the static conditions of the tank test. Additionally, differences in the initiation time of the tests exert a substantial influence on the results. These observations suggest that the conceptual framework developed for the DSLT cannot be directly applied to the injection percolation test. Therefore, the DSLT was selected as the leaching procedure for this study.

The primary objective of this research was to improve the understanding and quantification of BPA leaching from sewer renovation products. These products are typically based on resins such as epoxy and vinyl ester, which are commonly used in rehabilitation materials like liner systems. Specifically, the aim was to define the test conditions for the Dynamic Surface Leaching Test (DSLT) and to evaluate the amount of BPA leached from these materials over time, taking into account their use under real-world conditions. The inclusion of bisphenol F (BPF) in the study is based on the observation that it exhibits properties similar to those of BPA and is increasingly used as a substitute monomer in plastic production (Moon, 2019).

1.5. Toxicity of Bisphenol A

Bisphenol A (BPA) has been identified as a reproductive toxicant and is classified as a Category 1B substance (EU (European Union, Commission Regulation) 2016). The World Health Organization (WHO) recognizes BPA as an endocrine disruptor, a substance capable of exerting hormone-like effects that interfere with hormonal systems in both humans and wildlife, thereby posing potential health risks. Studies have demonstrated that exposure to BPA can lead to various adverse effects, including feminization and limb malformations in fish (UBA (Umweltbundesamt) 2010). Additionally, research has shown that male deer mice exhibit feminized behavior and avoid female conspecifics following BPA exposure (Jašarević et al., 2011). In response to these

Table 1
Overview of limit and guideline values for BPA.

Rules/Source	Value	Notes
Trinkwasserverodnung (TrinkwV), Germany (2023) (TrinkwV (Trinkwasserverordnung Germany) 2023)	2,5 μg/L	Limit value for drinking water
European Parliament (EP) directive proposal (water intended for human consumption of the EU, 2018) (EP (European Parliament) 2018)	0,01 μg/L	WHO recommendation and also proposed by the EU-Commission; the value was not included in the EU-guideline in 2020 (EP (European Parliament) 2020).
EP directive proposal (water policy, protection of groundwater and surface water, 2022), updated based on European Food Safety Authority (EFSA, 2023) (EFSA (European Food Safety Authority) 2023)	$1{,}7 \cdot 10^{-4}$ μg/L	Environmental Quality Standard (EQS) for surface water (original value: 3,4 10^{-5} μg/L; increased by a factor of 5 by adjusting the TDI) (EP (European Parliament) 2024)
Insignificance thresholds for groundwater by LAWA (Bund-/ Länderarbeitsgemeinschaft Wasser Germany, 2016) (LAWA (Bund-/Länderarbeitsgemeinschaft Wasser Germany) 2016)	–	For the OgewV (Verordnung zum Schutz der Oberflächengewässer, Germany 2016), the list of substances would need to be adjusted accordingly, when the EQS regulation is revised to include BPA (OgewV (Verordnung zum Schutz der Oberflächengewässer Germany) 2016).
U.S. FDA (Food and Drug Administration of the United States)	-	Although concerns exist regarding BPA in food packaging, drinking water regulations currently do not specify a limit.

findings, Denmark, Canada, and several U.S. states took precautionary measures in 2008 by banning the use of BPA in products frequently used by children, such as baby bottles. BPA can enter the human body not only through ingestion of contaminated food but also via oral and dermal exposure from sources such as thermal paper and cosmetics [(UBA (Umweltbundesamt) 2010, Jašarević et al., 2011)]. (Rudawska et al., 2022) concluded that cured epoxy resins do not exhibit cytotoxic effects on living cells, even upon contact with human cells. However, there is limited consensus regarding the full extent of BPA's harmful effects and those of its alternatives which has led to discrepancies in regulatory limits and guideline values across various jurisdictions. These differences can be attributed to divergent scientific interpretations, as well as national or regional variations in data assessment and safety standard development. Table 1 presents a comparative analysis of selected regulations and guidelines, highlighting the variability in threshold values set by different regulatory bodies or regions.

Furthermore, the toxicological effects of the chemical mixture present in the eluates from resin leaching were examined using ERα-CALUX analysis (Estrogen Receptor alpha responsive Chemical Activated LUciferase gene eXpression). This analysis aimed to assess the overall estrogenic activity of the complete eluate, rather than focusing solely on BPA, as is the case with BPA-R bioreporter assays (Rajasärkkä and Virta, 2013).

1.6. Installation of liners for sewer renovation

Two methods are commonly used for installing resin-soaked inliners in renovated sewer, drinking water, or wastewater pipes. The most widely employed technique by companies and industrial partners is the inversion method, which involves attaching an inversion tube to the pipe inlet. This method is frequently used on residential sewer lines in conjunction with epoxy resin. After the inversion tube is installed, a

Table 2
Installation methods of sewer rehabilitation products (RSV (Rohrleitungssanierungsverband) 2021).

	Needle felt liner	Glass fiber reinforced needle felt	Fiberglass liner
DN-Area* in mm	DN 100-DN 2200	DN 100-DN 1600	DN 150-DN 2000
Composite thickness in mm*	3 to 50	3 to 40	3 to 30
Resin type**	EP, UP, VE	EP, UP, VE	UP, VE
Installation procedure	Inversion, Combination Moving-in / Inversion		Moving-In, Inversion
Hardening process	Heat curing (water/steam) Ambient temperature hardening at small DN		UV-curing, combination hardening
Bendability (Radius dependent) *	≤ 45° (Larger arcs with radii ≥ 5D possible with restrictions)		≤ 15°

* The values mentioned are possible areas of application based on manufacturer information.
** EP – Epoxy resin, UP – unsaturated polyester resin, VE- vinyl ester resin

resin-soaked felt hose, twisted onto a rotating drum, is inserted into the pipe using warm compressed air. Epoxy resins undergo a chemical reaction that leads to hardening when exposed to heat; however, this reaction should be controlled and accelerated by the application of additional heat (RSV (Rohrleitungssanierungsverband) 2021). During inversion, the side of the felt originally facing inward is inverted to come into direct contact with the old pipe. Ensuring a proper bond between the new resin system and the existing pipe is critical to prevent the formation of open spaces. Such gaps can allow water to infiltrate and escape uncontrollably through leaks in the old pipe, thereby compromising the intended function of the new resin system. This back migration is particularly common when using a preliner film, which is primarily applied in pipes located in groundwater areas. As an alternative to inversion, a sliding hose method can be employed. In this approach, a resin-soaked hose liner is pulled into the bottom of the old pipe using a hook, then inflated with compressed air along the pipe length to be rehabilitated. The UV-CIPP (Cured-In-Place Pipe) technology utilizes photocatalysts embedded within the resin, which are activated by ultraviolet light emitted from a sliding device known as the UV lamp chain. This device moves systematically along the renovated pipe section to ensure uniform curing. Table 2 presents a set of guidelines for selecting appropriate liners.

For pipes with a nominal diameter of DN 800 (approximately 800 millimeters or 31.5 inches) and larger, the inversion method is no longer the preferred installation technique. Instead, the sliding hose method has become the standard, as larger-diameter pipes require a more robust and controlled installation process to ensure the structural integrity and quality of the liner. In this application, the liner can be pre-manufactured in the factory and impregnated with the appropriate resin system. Furthermore, the use of a preliner can prevent direct contact between substances and the surrounding soil or groundwater.

2. Materials and methods

2.1. Resin samples and their productions

In the present study, four materials, each from a different German manufacturer with extensive experience in sewer rehabilitation, were selected for investigation. These materials comprised two epoxy resins and two vinyl ester resins. For the fabrication of the epoxy resin systems, silicone molds were employed, with the epoxy resin curing without bonding to the silicone mold due to its cross-linking properties. Both epoxy resin systems were successfully demolded without difficulty. However, the use of petri dishes as molds proved impractical for production, as these vessels were prone to rupture due to thermal expansion

Table 3
Test Matrix for the BPA and BPF investigations (sewer renovation products).

Leaching Medium	Age d	R1 (Epoxy resin with active monomer addition)	R2 (Epoxy resin without active monomer addition)	R3 (Vinyl ester resin, BPA content unknown)	R4 (Vinyl ester resin, BPA content unknown)
Deionized water	1	[●●●]	-	[●●●]	-
	7	●●●	●●●	●●●	●●●
	28	[●●●]	-	[●●●]	-

[●●●]: Analysis of bisphenols in the mixed eluate; ●●●: Analysis of the bisphenols in the individual eluates;
-: not investigated

during curing. In contrast, the use of petri dishes for the two vinyl ester resin systems did not present any issues. According to manufacturer information, one epoxy resin (R1) contains an active BPA additive, while the other (R2) does not. Both epoxy resin systems, R1 and R2, are commonly used for the rehabilitation of corroded domestic sewage pipes. The manufacturers of the vinyl ester resins did not provide information regarding their BPA content. Industrial partners and resin component suppliers indicated that the actual monomer content could not be ascertained. Vinyl ester resin R3 is reported by its manufacturer to be styrene-free, whereas R4 contains styrene; both are used for renovating drinking water pipes. All four resins were subjected to leaching tests using deionized water, with the pre-storage time prior to elution as a key variable. For two resins (R1 and R3), sample ages of 1 day, 7 days, and 28 days were considered. Resins R2 and R4 were tested at a sample age of 7 days. The eluates from the Dynamic Surface Leaching Tests (DSLTs) conducted at these sample ages were analyzed individually to assess variability and reproducibility. An overview of the tests conducted is provided in Table 3.

The production of vinyl ester resin R3 samples involved a two-layer method. The initial 3 mm layer of vinyl ester resin was carefully applied to the mold and subsequently cured using UV light. Thereafter, the second layer was applied, followed by a second curing step for both layers. Notably, this resin system was produced without the incorporation of a hardener to prevent complete curing of the vinyl ester resin in a single pass. The presence of air pockets during the two-layer production process is a well-documented source of error in DSLT investigations (Fig. 1).

2.2. Dynamic Surface Leaching Test (DSLT)

The leaching process was conducted in accordance with the provisions of EN 16637-2:2023, employing the Dynamic Surface Leaching Test (DSLT), as schematically illustrated in Fig. 2.

Following a rigorous curing process and preliminary storage in a darkened environment, three test specimens, each with a diameter of 11 cm, a thickness of 5 mm, and a single surface area of 207 cm^2 (total surface area 621 cm^2 or 0.0621 m^2), were meticulously positioned on a stainless-steel holder. The specimens were then immersed in a colorless, transparent glass cylinder containing the leaching medium. The experiments utilized deionized water (ISO 3696:1987) produced by a Milli-Q EQ 7008 system (Merck KGaA). A water volume of 1.244 L was employed, resulting in a water-to-surface ratio of 20 L/m^2. This ratio, along with the experimental temperature of 20 °C, is based on standards adopted by the German Institute for Building Technology for the approval of resin systems used in pipe liners for sewer rehabilitation. The eluate-to-surface area ratio depends on the required volume, with permissible values ranging from 20 to 25 L/m^2. The glass vessel was sealed airtight and enclosed in a cardboard box during the leaching process to prevent photodegradation.

In accordance with the provisions of EN 16637-2:2023, eluate exchange intervals were set at 0.25, 1, 2.25, 4, 9, 16, 36, and 64 days.

2.3. Parameters

The following eluate parameters were determined:

- pH value and electrical conductivity (according to ISO 10523:2008-12 and ISO 7888:1985-05)
- DOC (according to EN 1484:1997-08)
- Concentrations of bisphenol A and F using GC-MS (according to EN ISO 18857-2:2012-01)
- GC/MS screening (according to DIN 3599:2022-02 and U.S. EPA, 2014: Method 8270E; SW-846) for further organic substances after the first elution step (6 hours) as a mixed eluate and after the seventh eluate removal (36 days) as a mixed eluate
- Screening for estrogenic effects using ERα-CALUX for the eluates from the last leaching step (64 days) as a mixed eluate (accredited to ISO17025 RvA L401)

The pH value and electrical conductivity were measured immediately after eluate collection. The primary focus of this research was on the monomers BPA (CAS number 80-05-7) and BPF (CAS number 620-92-8), which were analyzed using gas chromatography coupled with mass spectrometry (TRACE 1300, Q Exactive GC Orbitrap, Thermo Scientific). Additionally, the eluates were screened for other potentially harmful components using the same GC/MS setup. Based on the measured concentrations, the cumulative release of the material parameters was calculated using equation (Eq. 1).

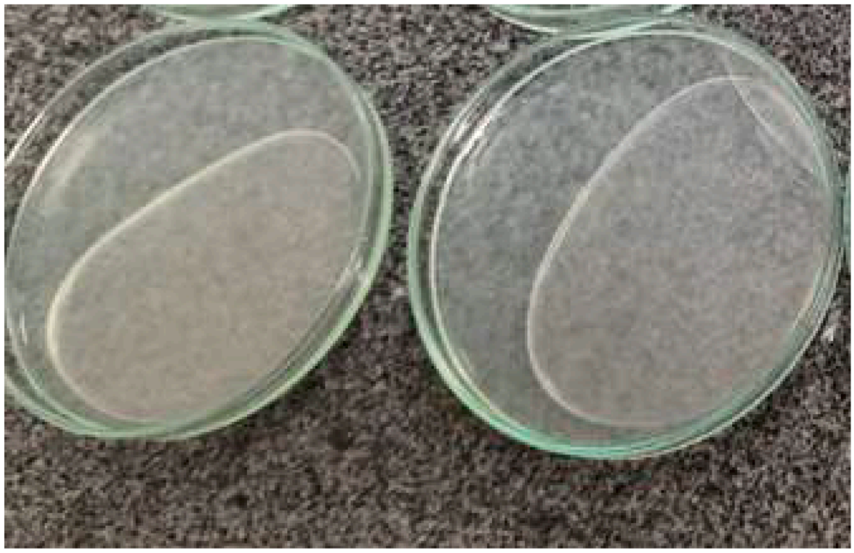


Fig. 1. Vinyl ester resin sample (R3) with air pockets between the layers when produced in petri dishes.

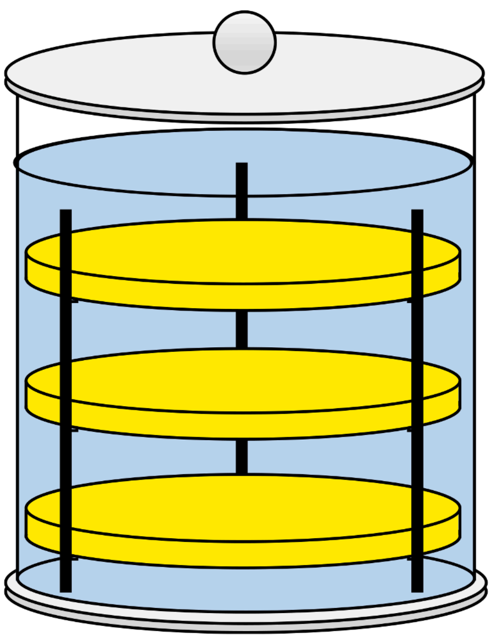


Fig. 2. Schematic set-up of the DSLT.

$$R_n = \sum_{i=1}^{n} R_i = \sum_{i=1}^{n} c_i \cdot \frac{L}{A} \tag{1}$$

R_n: cumulative release at the end of interval “n” in mg/m^2
R_i: Release during the leaching interval “i” in mg/m^2
c_i: concentration in eluate “i” in mg/l
L/A: Ratio of the eluent volume to the surface of the test specimen, L/A = 20 l/m^2

Note: The increased surface area of R3 samples could not be recorded and was therefore not taken into account. The calculated releases are therefore probably too high.

In accordance with DIN 3599:2022-02, a semi-quantitative analysis was conducted as part of this research. The estrogenic effects of the monomers bisphenol A and bisphenol F in the eluates were examined using the ERα-CALUX test, a method recognized as highly sensitive and effective (Gehrmann et al., 2018). Following analysis, the results were expressed as 17β-estradiol equivalents per liter (EEQ/L). Estradiol, the most potent naturally occurring estrogen, is often abbreviated as E2. Its chemical formula is $C_{18}H_{24}O_2$, and its CAS number is 50-28-2.

3. Results

3.1. pH, electrical Conductivity, BPA, BPF, and DOC

The blank values for pH and electrical conductivity were indicative of demineralized water. BPA was detected in the blanks of all experiments, with concentrations ranging from 0.006 to 0.11 μg/L. BPF was generally not detected in the blanks; however, quantifiable concentrations up to 0.005 μg/L were occasionally observed. It is important to note that the reported results for BPA and BPF do not include blank value contributions. The dissolved organic carbon (DOC) in the blanks was generally below the limit of quantification (LOQ) of 1 mg/L, except for one eluate with a concentration of 1.7 mg/L, likely due to contamination. Sulfate and chloride ion contents in the deionized water were determined by ion chromatography, with both ions found at concentrations below 0.1 mg/L. This assessment was conducted to exclude any potential interaction of these ions with BPA during leaching (Viñas et al., 2013, Ho et al., 2017). The four resins exhibited varied leaching behaviors. Epoxy resin R1 had negligible impact on the pH of deionized water, maintaining a standard value of approximately six. In contrast, epoxy resin R2 caused a pronounced alkaline shift, especially during the initial phase of the leaching test. Electrical conductivity measurements corroborated these findings: resin R1 exhibited minimal leaching, whereas the resins inducing a pH shift showed increased conductivity. Depending on the leached substances, DOC can also affect conductivity. Resin R2 displayed a pronounced wash-off effect, evident in the electrical conductivity profile (Fig. 3). The resin disks from all formulations showed no visible residues, such as biofilms or flocculated substances, between eluate replacements.

Vinyl ester resin R3 exhibited an acidic response, with the intensity increasing when leaching began at earlier time points. In contrast, R4 caused an increase in pH, but only during the later stages of the elution process, as confirmed by the electrical conductivity data (Fig. 4).

Quantification of DOC leaching was not feasible for resin R1. For resin R4, only a limited number of measured values exceeded the limit of quantification. Consequently, Fig. 5 presents the cumulative DOC release (as defined in Eq. 1) for resins R2 and R3 only. It is evident that resin R3, in particular, releases substantial amounts of DOC, and that the

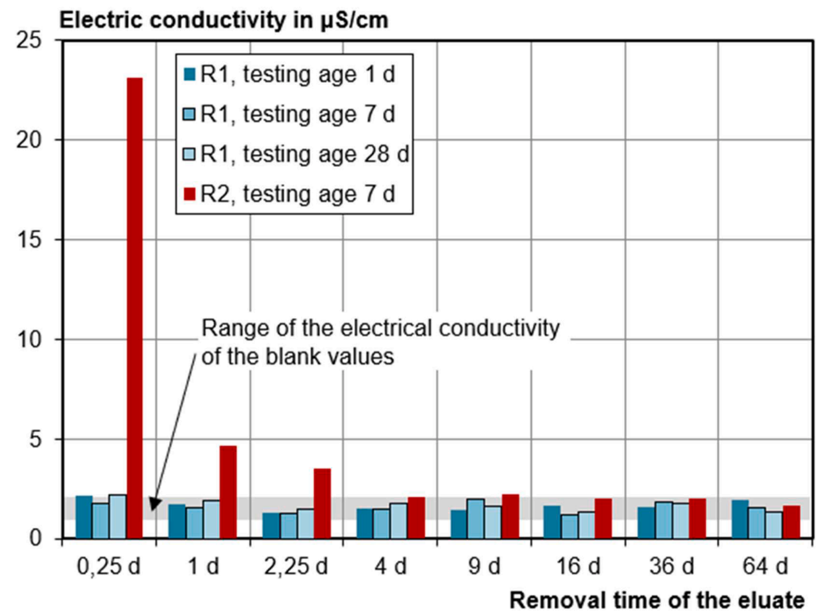


Fig. 3. Electrical conductivities in the DSLT with deionized water compared to the blank value (epoxy resins for sewer renovation products).

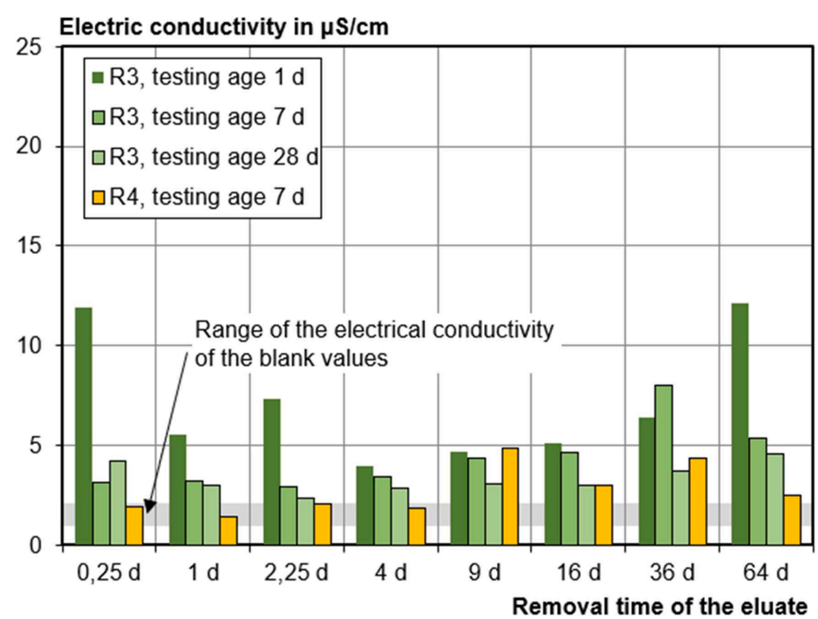


Fig. 4. Compilation of the electrical conductivities in the DSLT with deionized water compared to the blank value (vinyl ester resins for sewer renovation products).

duration of pre-storage has a significant influence on the extent of release. Earlier initiation of the leaching test leads to higher DOC levels. At a test age of seven days, the DOC release from R3 is reduced by 18 % compared to that observed when the sample age is one day. The release values shown in this figure are notably elevated and cannot be explained by the identified substances alone.

The BPA concentrations for resins R2 and R4 show only a marginal increase relative to the blank values, which may potentially compromise the validity of the results. The calculated cumulative release is presented in Fig. 6.

Resin R3 exhibited a consistent release of both BPA and BPF, likely due to the two-layer construction of the samples, as previously described. During the experiment, air pockets between the layers were partially filled with water, leading to an increase in the wetted surface area. This, in turn, resulted in elevated concentrations of the substances in the eluate during the leaching test. The cumulative release of BPA for resin systems R3 and R4 is shown in Fig. 7.

A comparison of the release profiles of BPA and BPF reveals that BPA was released in significantly higher amounts, by a factor of 18 to 26 after 64 days. This finding suggests the potential for BPA contamination in commercially available products marketed as containing BPF. However, the active addition of BPF to the examined products appears unlikely, as this monomer was not leached from resin R1 in quantifiable concentrations. Consequently, Fig. 8 presents only the three resin systems that have not yet been discussed in relation to bisphenol F leaching.

As schematically illustrated in Table 3, no mixed eluate was analyzed for the seven-day sample age, in contrast to the sample ages of one and 28 days. However, the eluates from each of the three replicate glass cylinders were analyzed individually for the monomers BPA and BPF. The mean values for this sample age are presented in Figs. 7 and 8. The

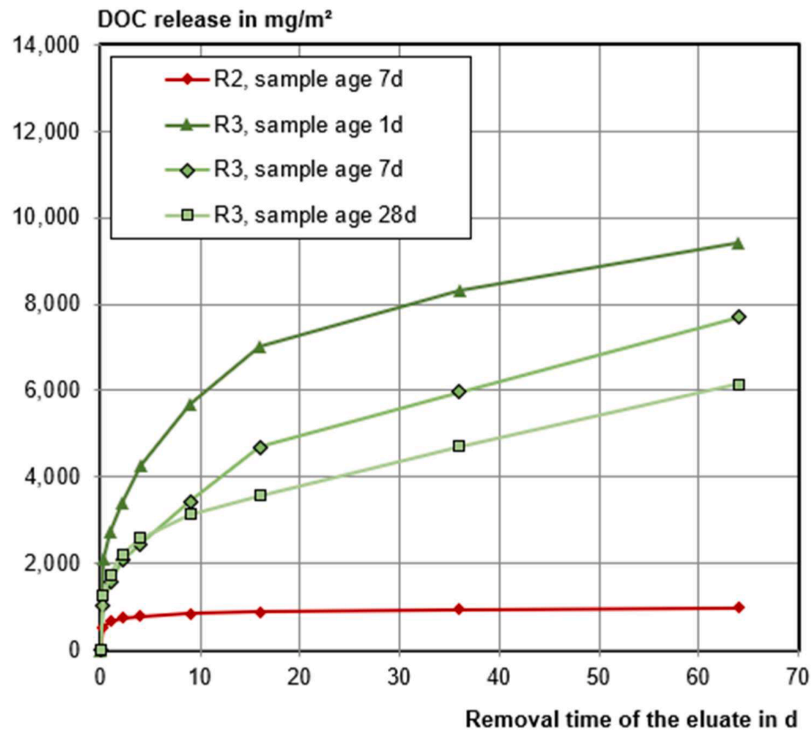


Fig. 5. DOC-release of the resin-systems R2 and R3.

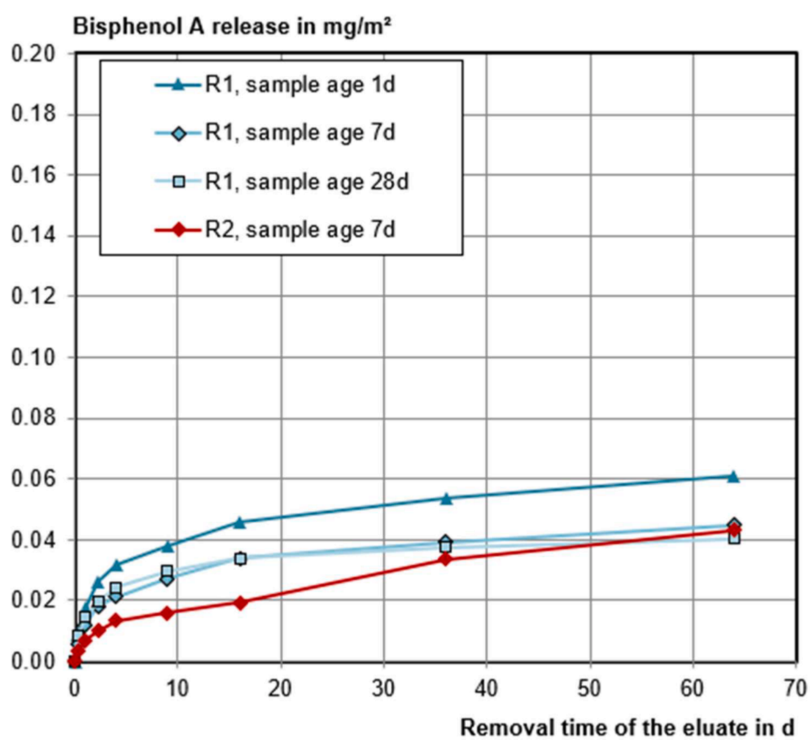


Fig. 6. Release of BPA (R1 and R2).

variation in cumulative release, defined by the minimum and maximum values across individual resin systems used for sewer rehabilitation, is negligibly small and is summarized in Table 4. Consequently, for future studies, it is considered acceptable to use only the mixed eluate from the leaching test when investigating organic parameters.

3.2. *GC/MS-Screening*

The substances listed in Table 5 were identified using a semi-quantitative GC/MS screening procedure in accordance with DIN 3599:2022-02 (DIN 3599 2022-02).

A range of concentrations was determined for the substances found in eluates leached from resins used in sewer renovation products, as

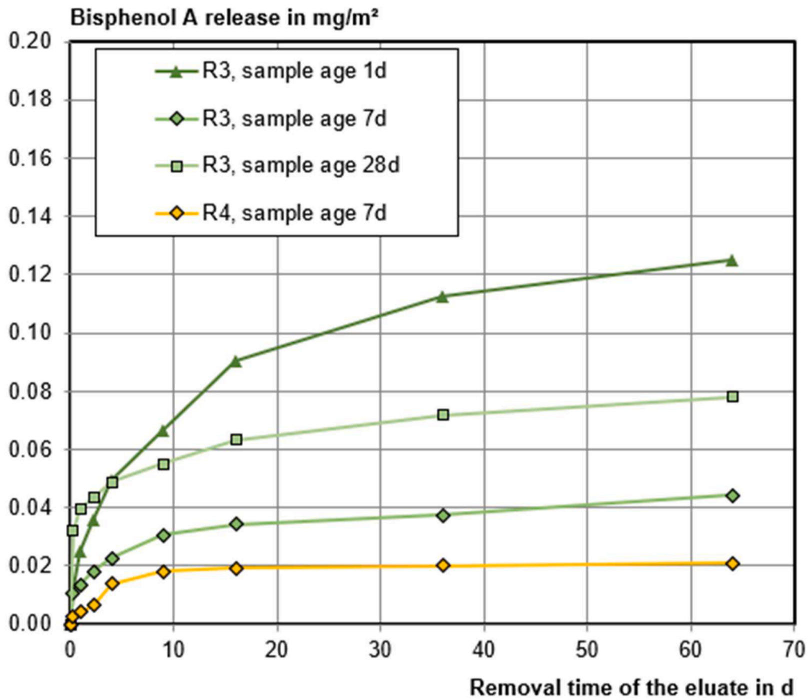


Fig. 7. Release of BPA (R3 and R4).

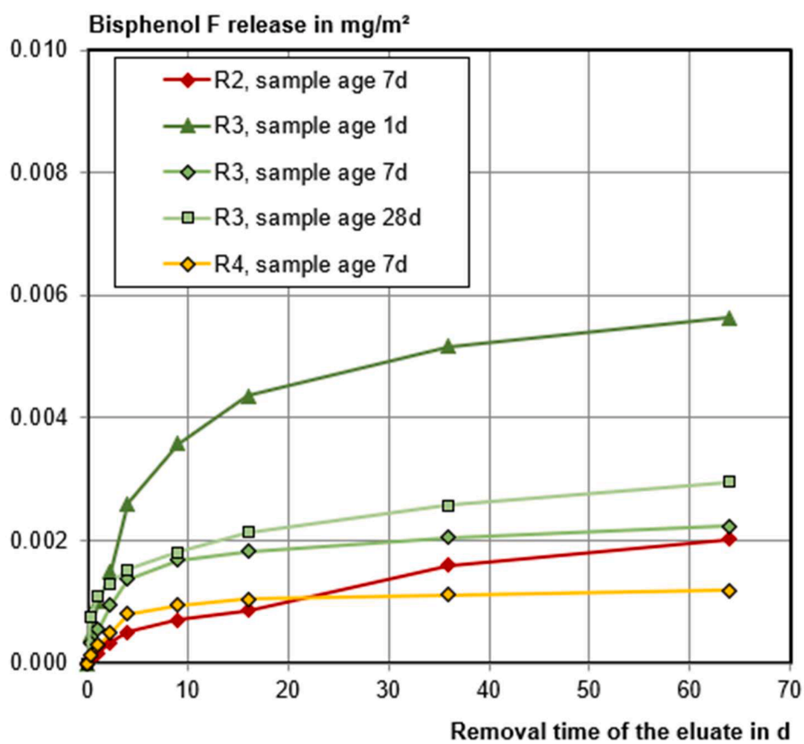


Fig. 8. Release of BPF from resins R2 to R4 with quantifiable eluate concentrations.

shown in Table 6, along with preliminary indications of their toxicity. It is important to note that the concentrations obtained from the semi-quantitative analysis should be considered reference values only. Accurate quantification requires the use of calibration standards; therefore, a reliable risk assessment cannot be conducted based on these data alone. Nonetheless, the analysis highlights the potential need for further research to assess whether regulatory modifications are warranted. Preliminary findings, based on the screening and quantification of both substance classes, indicate that further investigation of phenolic compounds and nitropyrazoles is justified.

Styrene was detected in the eluates of R4 after the first and seventh extraction steps, with a concentration of 13 µg/L. The other listed substances, each represented by a single value, were detected only once during the semi-quantitative GC/MS screening.

Table 4
Comparison of mean values and minimum/maximum cumulative releases of BPA and BPF in the Dynamic Surface Leaching Test (DSLT) for epoxy and vinyl ester resins used in sewer renovation, with a sample age of seven days.

Sample	Cumulative release R64d					
	mean BPA mg/m^2	minimum BPA	maximum BPA	mean BPF	minimum BPF	maximum BPF
R1 (7d)	0.045	0.035	0.050	0.0002	0.0002	0.0002
R2 (7d)	0.043	0.039	0.048	0.0020	0.0017	0.0023
R3* (7d)	0.044	0.043	0.045	0.0022	0.0022	0.0022
R4 (7d)	0.021	0.020	0.021	0.0012	0.0011	0.0012

* The increased releases may be attributed to the two-layer construction of the test specimens, which allowed water to penetrate the material and thereby increased the effective leached surface area.

3.3. ERα-CALUX

As described in the Materials and Methods section, the hormonal effect of eluates from sewer renovation products was assessed using the ERα-CALUX test. The final eluates after 64 days were analyzed as mixed samples, and all results of this investigation are presented in Table 7. The quantification limit in each case depended on the volume of the enriched sample used but was always at least 200 milliliters.

To ascertain the risk potential for organisms, effect-based trigger (EBT) values were introduced. According to van der Oost et al. (van der Oost et al., 2017), a potential risk to humans is characterized by a value of 0.5 ng E2 eq./L. Brand et al. (Brand et al., 2013) suggested an ERα-CALUX value of 3.8 ng E2 eq./L as an EBT, indicating the potential for adverse effects on hormonal balance. In alignment with the EU Commission's proposal, the German Federal Environment Agency (UBA) has recommended a threshold value of 0.4 ng E2 eq./L, proposing its inclusion in the 'watch list' for hormonally active substances. It was found that the UBA limit of 0.4 ng E2 eq./L was exceeded in only one instance. Only the resin samples R1 (7 days) and R4 exhibited a potentially problematic effect. Resin R4 demonstrated the highest value in the ERα-CALUX analysis following the final eluate extraction, with 0.47 ng 17β-estradiol/L, despite having the lowest cumulative BPA release. Consequently, it can be inferred that the estrogenic effect of this eluate is attributable to other substances. One potential contributor to the observed hormonal effect is styrene, detected by GC/MS screening in the eluates of this resin system; this substance is classified as a reproductive toxicant by the European Chemicals Agency (ECHA). Another possible contributor is 4-Methyltetrahydro-2H-pyran-2-one, whose water-polluting potential remains unclassified. The impact of additional substances on the hormonal effect was most pronounced in this resin system. However, it is noteworthy that all samples comply with the proposed limit values.

4. Discussion

The evaluation concept for sewer renovations, developed in a research project (Brameshuber and Vollpracht, 2011), is employed to classify the releases determined in leaching tests. This concept, created for the German Institute for Building Technology, involves evaluating predicted concentrations in groundwater outside the pipe, disregarding any potential entry into the environment via water inside the sewer (e.g., during rainwater sewer renovations). The interaction between groundwater and resin is limited to damaged areas of the canal, resulting in a relatively small contact area. Brameshuber and Vollpracht (Brameshuber and Vollpracht, 2011) defined the application scenario as a 100-meter-long canal section with a diameter of 1.2 meters. The canal consists of segments four meters in length, connected by joints 5 mm thick (Fig. 9). These joints were designated as damaged areas, and it was assumed that the joint material was entirely absent.

The model's transfer function establishes a relationship between releases measured in the DSLT and concentrations in groundwater at the point of compliance. The point of compliance is defined as the last joint, with groundwater concentrations averaged over a distance of two meters from the pipe and over a period of six months. It is important to note that the model incorporates several simplifications. For example, it assumes that the released substances are neither sorbed nor degraded in water-saturated soil, which may lead to an overestimation of actual concentrations for many compounds. Despite this limitation, the approach offers the advantage that the calculated groundwater concentration is independent of the properties of the leached substance, ensuring a consistent correlation across all compounds. The transfer function, a crucial component of the model, was derived in (Ahrens and Vollpracht, 2024) and is illustrated in Fig. 10.

The mean concentration of a given substance in groundwater at the point of compliance should not exceed the established limit values. In Germany, threshold values for groundwater, known as Geringfügigkeitsschwellen (GFS), have been defined. The GFS values represent concentrations below which contamination by a particular substance is considered negligible. Using the GFS values and the equation presented in Fig. 10, the permissible release in the Dynamic Surface Leaching Test (DSLT) can be calculated as follows:

$$permR_{64d} = GFS/0.00199 = 502 \cdot GFS \quad (2)$$

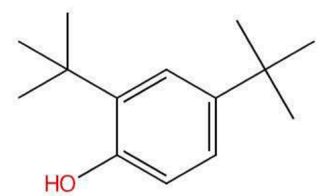
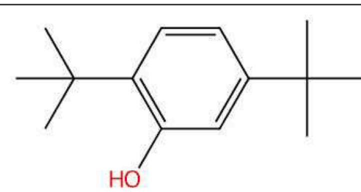
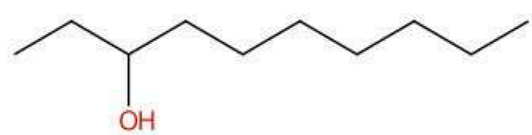
GFS: insignificance threshold in μg/l

It should be noted that no GFS values currently exist for BPA or other bisphenols. However, using the Environmental Quality Standard (EQS) for surface water of 1.7×10^{-4} μg/L (see Table 1), an upper limit for BPA release of 0.085 mg/m^2 is calculated based on the model described above.

Notably, diffusion- and depletion-controlled leaching behavior were not observed during the leaching test. The DSLT is designed to elicit diffusion-controlled leaching. In the absence of such behavior, alternative leaching regimes, such as daily or weekly medium changes, would produce different release profiles. Literature reports indicate non-diffusion-controlled release of the monomer BPA at selected sampling times over several weeks (Bruchet et al., 2014), and BPA leaching from plastic materials has been shown to increase with storage time (Uadia et al., 2019). It is acknowledged that other release patterns may occur in real-world applications, and that this evaluation does not fully capture the environmental impact of BPA leached from these resins. Rather, it should be considered a standardized approach. Nevertheless, this method enables a comparative analysis of the resins. Based on the derived limit value, an initial assessment can be performed. Table 8 summarizes the total releases, with any exceedances of the proposed R_{64d} (EQS) limit of 0.085 mg/m^2 highlighted in bold.

It is imperative to implement a rigorous and meticulous monitoring process to identify potential sources of error, such as the presence of air pockets, which have been observed to increase the effective leached surface area. Such measures will ensure the successful continuation of the leaching test without premature termination. Consequently, the BPA concentration can be recalculated based on a water-to-surface ratio of 20 L/m^2. It is essential to account for the additional leached area in the R3

Table 5
Identified substances in eluates after leaching from sewer rehabilitation products.

Compound	CAS No.	Substance group	Structural formula
2,4-Di-tert-butylphenol	96-76-4	Phenols	
2,5-Di-tert-butylphenol	5875-45-6	Phenols	
3-Decanol	1565-81-7	Aliphatic Alcohol	

(continued on next page)

Table 5 (*continued*)

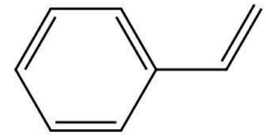
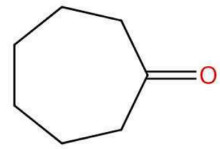
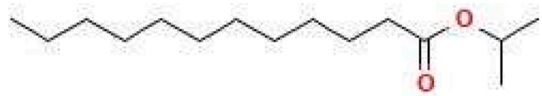
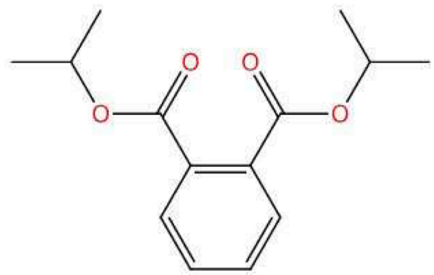
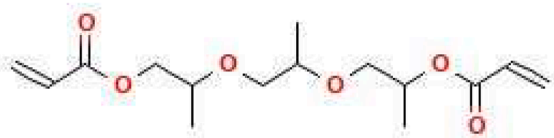
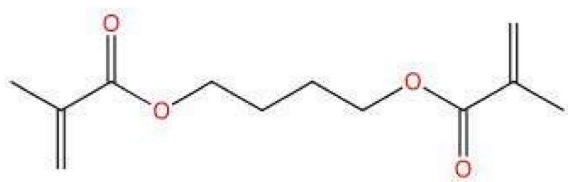
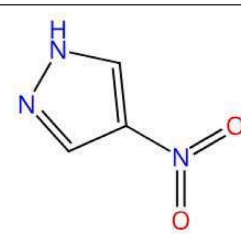
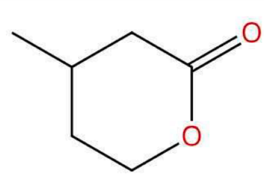
Styrene	100-42-5	Aromatic hydrocarbon	
2-Methylhexacosane	1561-02-0	Aliphatic hydrocarbon	
Cycloheptanone	502-42-1	Ketone	
Isopropyl laurate	10233-13-3	Fatty acid esters	
Diisopropyl phthalate	605-45-8	Phthalates	
Tripropylene glycol diacrylate	42978-66-5	Phthalates	
Butanediol dimethacrylate	2082-81-7	Acrylate	
4-Nitropyrazole	2075-46-9	Pyrazole	
2H-Pyran-2-one, tetrahydro-4-methyl-	1121-84-2	Pyrans	

Table 6
Source information for identified substances based on data from GESTIS (Gefahrstoffinformationssystem, Germany) (GESTIS) and the European Chemicals Agency (ECHA) (ECHA).

Compound	Concentration range of semi-quantitative Analyzes	Resin	Selected information from the GESTIS Substance Database and the European Chemicals Agency (ECHA) information page
2,4-Di-tert-butylphenol	20-340 μg/L	R1	- acute or chronic health risks - highly hazardous to water
2,5-Di-tert-butylphenol	270-305 μg/L	R2	- No classifications yet, possibly carcinogenic, mutagenic and/or toxic for reproduction
3-Decanol	1.4-3.2 μg/L	R3	- No classifications yet, possibly carcinogenic, mutagenic and/or toxic for reproduction
Styrene	13 μg/L	R4	- Risk of reproductive toxicity suspected clearly - hazardous to water
2-Methylhexacosane	78-111 μg/L	R1	- no classifications yet
Cycloheptanone	0.29 μg/L	R3	- acute or chronic health risks - no classification for water-endangering effects
Isopropyl laurate	46-53 μg/L	R1, R2	- low acute toxicity - generally hazardous to water
Diisopropyl phthalate	17 μg/L	R2	- No classifications yet, possibly carcinogenic, mutagenic and/or toxic for reproduction
Tripropylene glycol diacrylate	0.29 μg/L	R3	- acute or chronic health risks - clearly hazardous to water
Butanediol dimethacrylate	1.3 μg/L	R3	- low systemic toxicity - slightly hazardous to water
4-Nitropyrazole	0.8-53 μg/L	R3	- acutely toxic - no classification for water-endangering effects
2H-Pyran-2-one, tetrahydro-4-methyl-	26 μg/L	R4	- No classifications yet

Table 7
Results of the ERα-CALUX test on the final eluate of the DSLT with deionized water.

Resin	Age (d)	Blank value (ng 17β-estradiol/L)	Test value (ng 17β-estradiol/L)
R1	1	< 0.071	< 0.180
	7	< 0.071	0.089
	28	< 0.071	< 0.120
R2	7	< 0.026	< 0.042
R3	1	< 0.056	< 0.053
	7	< 0.056	< 0.050
	28	< 0.056	< 0.047
R4	7	< 0.049	0.470

resin system caused by the opening of air pockets formed during manufacturing, in order to apply a correction factor for the skewed leaching data. Assuming the additional leached area corresponds to half of a complete resin disk surface (see Fig. 1), this area is 95 cm^2. The water-to-surface ratio should then be corrected from 13.67 L/m^2, corresponding to a cumulative BPA release of 0.125 mg/m^2 for resin system R3 at a sample age of one day, to 20 L/m^2 with a correction factor of 0.685.

The adjusted cumulative BPA release is therefore 0.0856 mg/m^2, marginally exceeding the proposed guideline value of 0.085 mg/m^2. This correction factor was also applied to calculate adjusted values for Dissolved Organic Carbon (DOC) and ERα-CALUX analysis (see Table 9).

As illustrated in Table 10, a statistical analysis was conducted to compare the resin systems for sewer rehabilitation supplied by various companies. Additionally, the standard deviation (SD) of the mean BPA and BPF release values is provided to facilitate comparison across different sample ages.

The distribution of the monomer BPF in the cumulative release of the eluates is found to be nearly equivalent to the mean value when comparing resin samples R1 and R3 at a sample age of one day. However, the concentration of BPF remains remarkably low relative to that of BPA. This observation is consistent with findings for the sample age of 28 days. Considering this, it is evident that BPF does not pose a significant risk in the context of sewer renovation products and does not exceed the calculated upper limit of 0.085 mg/m^2 established by the Dynamic Surface Leaching Test (DSLT). It is imperative that future investigations establish an appropriate release limit for BPF in the DSLT. Additionally, it is essential to clarify the extent to which BPF may substitute for BPA in resin systems and the implications this substitution has for the toxicity of the eluate. The scatter in BPA concentrations across all resin systems is considerably lower than that for BPF. Specifically, based on the cumulative BPA release detected in eluates from all four resin systems for sewer renovation at a sample age of seven days, a scatter of only 28.16 % around the mean was calculated. This finding is notable, especially given that the exact BPA content in the resin systems is not fully known and was not disclosed by manufacturers due to confidentiality.

All other release values are close to the permissible release indicated by the model, suggesting that commercial products are generally capable of meeting the stipulated upper limit for BPA release in the DSLT for sewer renovation products. However, it is important to note that there should not be a significant absence of joint material in a pipe section beyond the model assumption of 0.5 m^2 over a 100-metre pipe length. The leaching behavior of the cured resin system was examined solely at 20 °C, as required by the approval regulations of the German Institute for Building Technology (DIBt). During liner operation, temperatures may vary, with lower values (e.g., 10 °C groundwater temperature) and higher values caused by warmer wastewater and/or solar heating, especially in summer. This temperature variability was not considered in the present study. Further research is necessary to explore the surface development of resin systems under varying temperatures and the resulting leaching mechanisms and released substances. There is a considerable need for experimental setups that account for additional parameters, including the aging of resin samples over extended periods (several years) and the subsequent release of chemical compounds. Nonetheless, the DSLT provides a satisfactory evaluation of the initial leaching behavior within a period of two months (64 days). Moreover, further studies are needed to assess the impact of microorganisms observed near sewage pipes in real-world conditions. Subsequent releases or development of substances due to wastewater interactions and their effects on ecosystems should also be examined (Yuan et al., 2024, Nielsen et al., 1992, Mattsson et al., 2015). As noted by (Makinwa and Uadia, 2017), rivers, estuaries, and lakes act as sinks for bisphenol A accumulation, a compound originating from sewage, plastic debris, and landfill waste. A significant increase in BPA leaching from polycarbonate tubes at 37 °C compared to 20 °C into sea and river water with varying pH levels was reported by (Sajiki and Yonekubo, 2003). Regular monitoring of BPA degradation and its substitutes under aerobic and anaerobic conditions in surface waters remains a critical area of research for regulatory bodies.

The BPA concentrations in the eluates of resin R1 (with a sample age of seven days) and R4, both of which approached or exceeded the

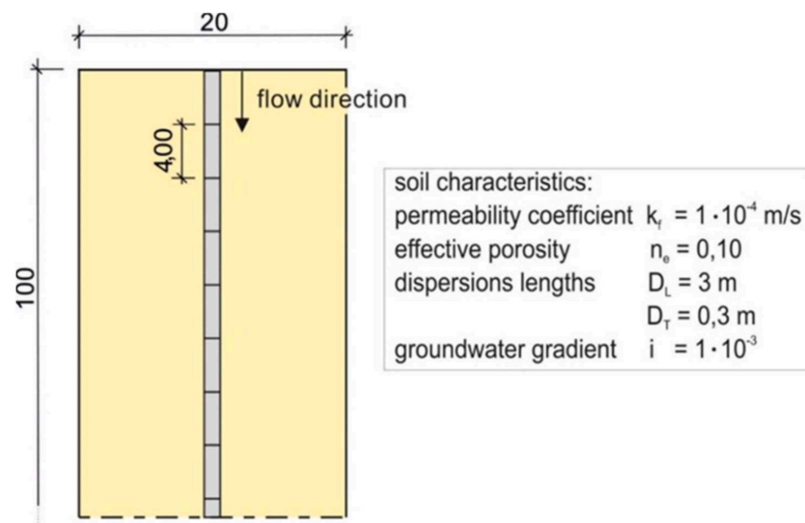


Fig. 9. Top view and section of the model area, dimensions in meters, adapted from (Brameshuber and Vollpracht, 2011).

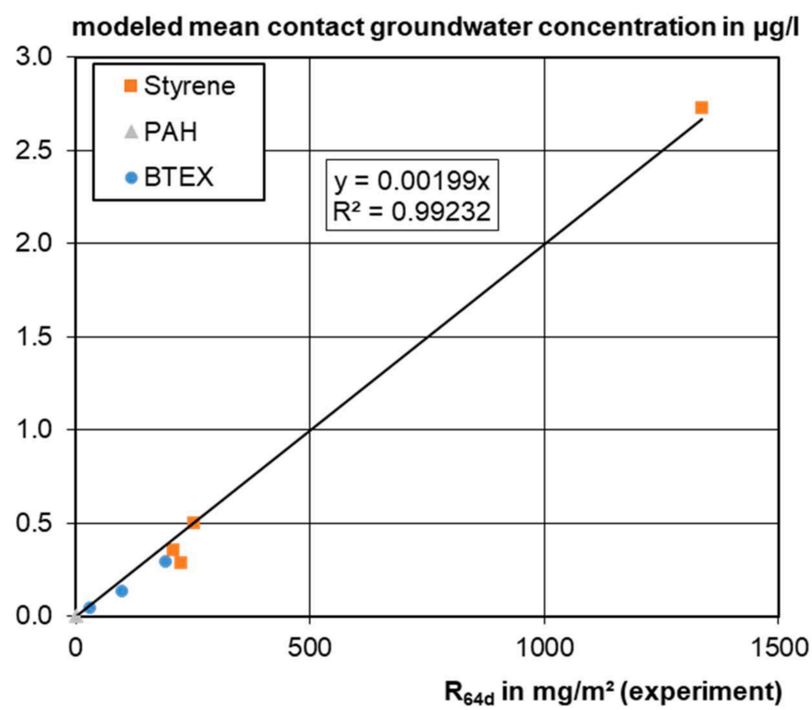


Fig. 10. Evaluation concept developed in (Brameshuber and Vollpracht, 2011): Functional connection between calculated mean contact groundwater concentration and release in the DSLT.

Table 8
Overview of the total releases determined for epoxy and vinyl ester resins. Values exceeding the upper limit of 0.085 mg/m², calculated based on the EQS, are shown in bold.

Leaching Medium	Parameter	Cumulative release R_{64d}							
		R1			R2, 7d	R3*			R4, 7d
		1d mg/m²	7d	28d	mg/m²	1d mg/m²	7d	28d	mg/m²
Deionized water	DOC	< 160	< 160	< 160	983	9426	7700	6140	175
	BPA	0.061	0.045	0.040	0.043	**0.125**	0.044	0.078	0.021
	BPF	< 0.0002	< 0.0002	< 0.0002	0.0020	0.0038	0.0015	0.0020	0.0012
	ERα- CALUX	ng 17β-estradiol/L							
		< 0.018	0.089	< 0.120	< 0.042	< 0.053	< 0.050	< 0.047	0.470

* The increased releases may be attributed to the two-layer construction of the test specimens, which allowed water to penetrate the material and thereby increased the effective leached surface area.

Table 9
Overview of the total releases determined for vinyl ester resin R3 with the applied correction factor. Values exceeding the upper limit of 0.085 mg/m², calculated based on the EQS, are highlighted in bold.

Leaching Medium	Parameter	R3		
		1d mg/m²	7d	28d
Deionized water	DOC	9426	5275	4206
	BPA	**0.086**	0.030	0.053
	BPF	0.0002	0.0010	0.0014
	ERα- CALUX	ng 17β-estradiol/L		
		< 0.036	< 0.034	< 0.032

proposed threshold values for estrogenic activity, as determined by the ERα-CALUX assay, remained within the permissible range calculated by the model and were lower compared to those in other eluates. Consequently, it can be concluded that the observed estrogenic effect is likely attributable to the presence of other substances, potentially in combination with bisphenols. The resin sample R1, aged for seven days, exhibited a semi-quantitative concentration of 340 µg/L 2,4-di-tert-butylphenol following the first and seventh eluate collection steps. This value exceeded that of the one-day-old sample, which measured 316 µg/L. Additionally, the seven-day-old R1 samples showed the presence of isopropyl laurate, with concentrations ranging from 46 to 47 µg/L. Resin R4 showed a styrene concentration of 13 µg/L in both eluates analyzed via semi-quantitative GC/MS screening, representing the highest values among all eluates examined. The analysis of the mixed eluate from the initial elution step of this formulation also revealed the presence of tetrahydro-4-methyl-2H-pyran-2-one at a concentration of 26 µg/L. As indicated by the aforementioned samples, BPF was detected only at minimal levels, frequently below the limit of quantification. The estrogenic effect observed in R4 may be attributable to the use of a hardener that was not employed in the R3 formulation.

5. Conclusion

The present study primarily examined the cumulative release of bisphenol A (BPA) at ambient temperature from resin systems used in sewer renovation products. Investigations into the leaching behavior of the monomer BPA, along with other organic parameters such as BPF, compounds identified through semi-quantitative GC/MS screening, and ERα-CALUX analytics, were carried out within the framework of the Dynamic Surface Leaching Test (DSLT), as outlined in EN 16637-2:2023. Deionized water was used as the testing medium to generate the eluates. Fig. 11 provides a schematic overview of the overall experimental approach and the various investigative steps applied to the resin systems evaluated in this study. This figure may also serve as a reference for future research in this field.

All epoxy and vinyl ester resin systems, regardless of sample age (one day, seven days, or 28 days), were found to comply with the stipulated maximum release value of 0.085 mg/m². This limit was derived using a model contact area and the insignificance threshold defined by the Environmental Quality Standard (EQS). Vinyl ester resin R3 exceeded this threshold when tested at a sample age of one day. As previously discussed, this result may be attributable to fabrication inaccuracies. The use of a two-layer specimen without a hardener is not recommended, as it may lead to an undesired increase in the leached surface area. The BPA release values obtained in the DSLT were close to the proposed limits, indicating that the contact area between the resin and groundwater should not exceed 0.5 m² over a 100-meter pipe length. If

Table 10
Statistical analysis for sewer renovation products (two epoxy and two vinyl ester resins).

Leaching Medium	Parameter	Comparison of the cumulative releases R_{64d}					
		R1, R3*				R1, R2, R3*, R4	
		1d (n=2)		28d (n=2)		7d (n=4)	
		mean mg/m²	SD	mean	SD	mean mg/m²	SD
Deionized water	BPA	0.0730	0.0120	0.0467	0.0067	0.0348	0.0098
	BPF	0.0020	0.0018	0.0011	0.0009	0.0012	0.0007

* The values calculated with the correction factor due to the increased leached surface area were used.

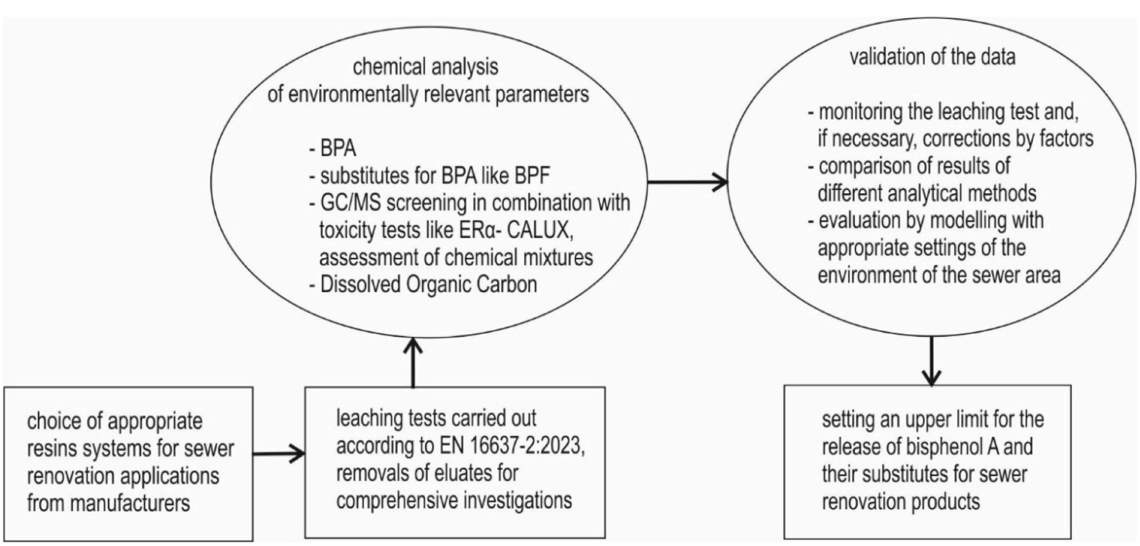


Fig. 11. Schematic overview of the approach used to investigate the leaching behaviour of sewer renovation products based on epoxy and vinyl ester resins using the Dynamic Surface Leaching Test (DSLT) in accordance with EN 16637-2:2023.

the World Health Organization's proposed precautionary value of 0.01 μg/L for BPA is applied to determine the allowable release in the DSLT, a threshold of 5.02 mg/m^2 would be calculated. Based on this limit, all resin systems tested would be considered compliant for both BPA and BPF. However, under scenarios involving more extensive damage, the predicted concentrations in groundwater would increase, necessitating a more detailed and realistic model. Such a model should account for degradation and/or sorption processes in the soil to provide a more accurate assessment of environmental risks associated with sewer renovation. It is also essential to highlight the current lack of research on BPA leaching during the initial week post-installation, particularly in real-world drinking water systems. This area should be prioritized in future investigations. The semi-quantitative gas chromatography/mass spectrometry (GC/MS) screening of eluates revealed the presence of substances whose toxicological and water-polluting potential warrants further study and, if necessary, regulatory classification. Moreover, the ERα-CALUX analysis indicated the potential presence of additional organic substances with hormonal and/or toxicological activity. Further investigations into these compounds as well as the effects of varying temperatures and pH levels of the leaching medium are strongly recommended.

CRediT authorship contribution statement

Konstantin Ahrens: Writing – original draft, Visualization, Validation, Project administration, Methodology, Investigation, Data curation, Conceptualization. **Reiner Gschwendtner:** Writing – review & editing, Validation, Investigation, Data curation. **Volker Linnemann:** Writing – review & editing, Project administration, Conceptualization. **Anya Vollpracht:** Writing – review & editing, Visualization, Supervision, Project administration, Funding acquisition, Conceptualization.

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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Data availability

Data will be made available on request.

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